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EU Wide Monitoring Survey on Waste Water Treatment Plant Effluents

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ACRONYMS

AA-EQS	Annual Average Environmental Quality Standard
ADBI	4-Acetyl-6-tert-butyl-1,1-dimethylindane, Celestolide®
AES	Atomic Emission Spectrometry
AHMI	6-Acetyl-1,1,2,3,3,5-hexamethylindane, Phantolide®
AHTN	6-Acetyl-1,1,2,4,4,7-hexamethyltetralin, Tonalide®
AITI	5-Acetyl-3-isopropyl-1,1,2,6-tetramethylindane, Traseolide®
AOPs	Advanced Oxidation Processes
BT	Benzotriazole
CID	Collision Induced Dissociation
DEET	N,N'-Diethyltoluamide
EC	European Commission
EDCs	Endocrine Disrupting Compounds
EDTA	Ethylenediaminetetraacetic acid
EEQ	Estrogen Equivalents
EHDPP	2-Ethylhexyldiphenylphosphate
ESI	Electrospray Ionisation
EU	European Union
GC	Gas Chromatography
GIS	Geographical Information System
HDPE	High Density Polyethylene
HHCB	1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexamethylcyclopenta(g)-2-benzopyran, Galaxolide®
HPLC	High Performance Liquid Chromatography
HRMS	High Resolution Mass Spectrometry
ICP	Inductively Coupled Plasma
IS	Internal Standard
Lab.	Laboratory
LAS	Linear Alkylbenzene Sulfonates
LC	Liquid Chromatography
LC-MS-MS	Liquid Chromatography (tandem) Triple Quadrupole Mass Spectrometry
LLE	Liquid-Liquid Extraction
LOD	Limit of Detection
LOQ	Limit of Quantification
MAC-EQS	Maximum Allowable Concentration EQS
MRM	Multiple Reaction Monitoring
MS	Mass Spectrometry
NSAID	Non-Steroidal Anti-inflammatory Drug
OPEs	Organophosphate Esters
PAC	Powdered Activated Carbon
PFASs	Perfluoroalkyl substances
PFBS	Perfluorobutane sulfonate
PFHxA	Perfluorohexanoic acid
PFHpA	Perfluoroheptanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctansulfonate
PNEC	Predicted No-Effect Concentration
POPs	Persistent Organic Pollutants
PPCPs	Pharmaceuticals and Personal Care Products
RP	Reversed-phase
RSD	Relative Standard Deviation
SDB	Styrol-divinylbenzene
SIM	Selected Ion Monitoring
SPE	Solid-Phase Extraction

TBP	Tributylphosphate
TBEP	Tris(2-butoxyethyl)phosphate
TCEP	Tris(2-chloroethyl)phosphate
TCP	Tricresylphosphate
TCCP	Tris(2-chloroisopropyl)phosphate
TDCP	Tris(1,3-dichloro-2-propyl)phosphate
TEHP	Tris(2-ethylhexyl)phosphate
TEQ _{bio}	Dioxin-like toxicity (toxic equivalents)
TIBP	Tri-iso-butylphosphate
TIC	Total Ion Current (chromatogram)
TPP	Triphenylphosphate
TT	Tolyltriazole
WFD	Water Framework Directive
WWTP	Waste Water Treatment Plant

LIST OF PARTICIPANTS

The following synopsis lists the participants in the exercise, who actively contributed to the project. The names of the persons listed may not be complete as many hands supported the project behind the scene. This important contribution is however gratefully acknowledged, too.

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SUMMARY

In the year 2010, effluents from 90 European waste water treatment plants (WWTPs) were collected and analysed in total for 160 organic chemicals and 20 inorganic trace elements. The analyses were complemented by applying also effect-based monitoring approaches aiming at estrogenicity and dioxin-like toxicity analysed by in vitro reporter gene bioassays, and yeast and diatom culture acute toxicity optical bioassays. The analytical work was performed in six European expert laboratories:

- Institute of Environment and Sustainability of the European Commission's Joint Research Centre (JRC) in Ispra, Italy
- IWW Water Centre, Mülheim a.d. Ruhr, Germany
- UMEÅ University, Umeå, Sweden
- RECETOX, Masaryk University, Brno, Czech Republik
- VITO, Mol, Belgium
- Federal Environmental Agency (Umweltbundesamt; UBA), Vienna, Austria

Analyses of organic substances were performed by solid-phase extraction (SPE) or liquid-liquid extraction (LLE) followed by liquid chromatography tandem mass spectrometry (LC-MS-MS) or gas chromatography high-resolution mass spectrometry (GC-HRMS). Inorganic compounds were analysed by inductively coupled plasma – mass spectrometry (ICP-MS) or ICP atomic emission spectrometry (ICP-AES).

This European-wide monitoring study on the occurrence of micropollutants in WWTP effluents represents the largest EU wide monitoring survey on WWTP effluents ever performed. It produced a comprehensive data set on many so far only locally investigated “emerging” compound classes including pharmaceuticals and personal care products (PPCPs), veterinary (antibiotic) drugs, perfluoroalkyl substances (PFASs), organophosphate ester flame retardants, pesticides (and some metabolites) or industrial chemicals such as benzotriazoles (corrosion inhibitors), polycyclic musk fragrances, x-ray contrast agents, Gadolinium compounds, and siloxanes. The obtained results show the presence of 131 target organic compounds in European wastewater effluents, in concentrations ranging from low nanograms to milligrams per liter. These results obtained from 90 different European WWTPs allow the calculation of a European median level for the chemicals investigated.

The most relevant compounds identified in the effluent water samples in terms of frequency of detection, maximum, average and median concentration levels were Sucralose, Acesulfame K (artificial sweeteners), PFOA, PFHxA, PFHpA, PFOS (perfluoroalkyl substances), N,N'-Diethyltoluamide (DEET; insect repellent), Benzotriazoles (corrosion inhibitors), the pharmaceuticals Bisoprolol, Carbamazepine, Ciprofloxacin, Citaprolam, Clindamycin, Codeine, Diltiazem, Diphenhydramin, Eprosartan, Fexofenadine, Flecainide, Gemfibrozil, Fluconazole, Haloperidol, Ibersartan,

Ibuprofen, Ketoprofen, Oxazepam, Risperidone, Sulfamethoxazole, Telmisartan, Tramadol, Trimethoprim, Venlafaxin, the organophosphate ester flame retardants Tri-isobutylphosphate (TIBP), Tributylphosphate (TBP), Tris(2-chloroethyl)phosphate (TCEP), Tris(2-chloroisopropyl)phosphate (TCPP), Tris(2-butoxyethyl)phosphate (TDCP), Tris(2-butoxyethyl)phosphate (TBEP), Triphenylphosphate (TPP), 2-Ethylhexyldiphenylphosphate (EHDPP), the x-ray contrast media Amidotrizoic acid, Iohexol, Iopromid, Iomeprol, Iopamidol, the pesticides Terbutylazine, Terbutylazine-desethyl (metabolite), MCPA, Mecoprop, Diuron, Triclosan (antibacterial), and Gadolinium (from magnetic resonance imaging contrast media used in hospitals).

The toxicity tests applied showed some toxicity. From the total number of 75 WWTP effluents analyzed for estrogenicity, 27 sample extracts showed estrogenic activity higher than the detection limit > 0.5 ng/l estrogen equivalents (EEQ). Twenty five effluent sample extracts we screened for dioxin-like activity, and twenty one out of these 25 tested sample extracts exceeded the detection limit (0.1 ng/l TEQ_{biotest}) but the maximal detected dioxin-like activity was only 0.4 ng/l TEQ_{biotest}. Finally, three out of 13 effluent samples tested on acute toxicity revealed themselves harmful for the growth of yeast and diatom organisms.

Finally, the results were compared with other published local, national, or smaller international studies on WWTPs. This comparison showed that lower median effluent concentrations were found in our large-scale European-wide study ($n=90$) compared to local studies of single WWTPs. It is difficult to compare results of median concentrations of one WWTP (or a small number) with the median of this study ($n=90$) because many different domestic and industrial plants around Europe were included. In contrast, maximum and average concentrations are in relatively good agreement to other studies.

What is clear is that the elimination of most of the anthropogenic substances in conventional WWTPs with secondary biological treatment is incomplete and improvements of wastewater treatment and subsequent treatments of the produced sludge are required to prevent the introduction of these micropollutants in the environment. It must be considered that today's conventional waste water treatment technology (mechanical and biological steps) is from the 1970s; it was designed to remove nitrogen and phosphorus, and some non-polar chemicals which are removed with the sewage sludge.

It is being discussed in Europe to upgrade WWTPs with additional tertiary or advanced treatment steps such as ozonation and/or powdered activated carbon adsorption to remove micropollutants from WWTP effluents. In the Swiss "Micropoll Strategy" project complementary treatment steps have been evaluated and it has been shown that water quality can be significantly improved using processes such as powdered activated carbon adsorption or ozonation. Also a further treatment by micro- or nanofiltration or reverse osmosis is possible for water reuse strategies.

Under the view of escalating population growth, and increased water stress in many regions of the world, reuse of treated water and waste water recycling are becoming more important options for water supply. The increasing worldwide contamination of freshwater systems with thousands of industrial and natural chemical compounds is one of the key environmental problems facing humanity. Although most of these compounds are present at low concentrations, many of them raise considerable toxicological concerns, particularly when present as components of complex mixtures (Schwarzenbach et al., 2006).

1. Introduction

Commission Directive 91/271/EEC (EC, 1991) concerns the collection, treatment and discharge of urban wastewater and the treatment and discharge of wastewater from certain industrial sectors. Its aim is to protect the environment from any adverse effects caused by the discharge of such waters. The increasing extent and level of municipal wastewater treatment in Europe in the past decades has significantly improved the quality of surface waters, even though obligations set for the European Union are not equally fulfilled by all its members (EC, 2004; Reemtsma et al., 2006). However, priority substances or other organic compounds are not regulated in waste water treatment plant (WWTP) effluents (EC, 1991), but in surface waters under the Water Framework Directive (EC, 2000).

Whilst household and industrial wastewater treatment has been implemented progressively across Europe, and existing treatment technologies produce water that meets current legislation on water-quality standards, it has been demonstrated that the removal of many emerging contaminants, including pharmaceuticals and personal-care products (PPCPs), hormones, and other industrial chemicals is incomplete, with many emerging contaminants being detected in treated effluents that are subsequently discharged to surface waters. Various studies over recent years have shown that treated municipal wastewater contributes significantly to water pollution from micropollutants (e.g.: Ashton et al., 2004; Castiglioni et al., 2006; Clara et al., 2005; De la Cruz et al., 2012; Gabet-Giraud et al., 2010; Gracia-Lor et al., 2010, 2012; Gros et al., 2010; Hollender et al., 2009; Jelic et al., 2012; Joss et al., 2005, 2006; Köck-Schulmeyer et al., 2011; Lindqvist et al., 2005; Martínez Bueno et al., 2012; Micropoll, 2011; 2012; Miège et al., 2009; Nakada et al., 2006; Paxéus, 2004; Radjenović et al., 2007a,b; Reemtsma et al., 2006; Ternes, 1998; Vieno et al., 2007; Zhang et al., 2008a).

Conventional WWTPs are designed to reduce loads of carbon, nitrogen, and phosphorus. In addition, non-polar chemical compounds are well removed by sorption. The most important removal pathways of organic compounds during wastewater treatment are biotransformation / biodegradation, abiotic removal by adsorption onto the sludge and stripping by aeration (volatilization) (Radjenović et al., 2007a). Several polar compounds, especially those which are poorly degradable, may however be discharged with WWTP effluents into receiving waters and then occur in surface waters (Reemtsma et al., 2006). Some polar chemical compounds such as Nonylphenol (Yu et al., 2009a; Zhang et al., 2008b) or Perfluoroalkyl Substances (PFASs) like Perfluorooctansulfonic acid (PFOS) and Perfluorooctanoic acid (PFOA) (Becker et al., 2008) are even formed in WWTPs from precursor compounds.

It is well established that insufficiently treated WWTP effluents are the main sources of PPCPs and other micropollutant residues in the aquatic environment, because many micropollutants are not considerably degraded in conventional WWTPs. Treated wastewater effluents are the main contributors to PPCPs and industrial chemicals loads in

rivers, as WWTP effluents are often major contributors to rivers' flows (Hollender et al., 2009; Kasprzyk-Hordern, et al., 2009).

Such discharges are near-continuous and hence these substances do not need to be persistent in nature to give rise to chronic effects due to long-term exposure (such as endocrine disruption) in aquatic ecosystems (EEA, 2011). However, veterinary pharmaceuticals residues are discharged also directly into the ecosystem via agricultural field run-off. In general, there are several different input pathways of micropollutants into surface waters: (1) Input with treated wastewater through WWTPs; (2) input with untreated wastewater through sewer overflows if the capacity of the sewer system or the wastewater treatment plant is exceeded; (3) input through leaks in the sewer network or wrongly connected sewers; (4) input of polluted rainwater from roofs and sealed areas through rainwater channels (Micropoll, 2011); and (5) other diffuse sources such as field run-off.

Moreover, there might be the possibility of contamination of water resources (surface and groundwaters) by the application of contaminated sewage sludge (solid waste of the wastewater treatment process) to soils or agricultural fields.

Mass balance calculations to estimate the fate of contaminants during waste water treatment, including sorption to sludge showed that usually less than 2% of the total mass load of pharmaceuticals is removed by sorption. For most pharmaceuticals (including Carbamazepine, Sulfamethoxazole, and Trimethoprim) adsorption to sludge is negligible. Some micropollutants, however, such as selected antibiotics (Ciprofloxacin, Norfloxacin), musk fragrances (Galaxolide[®], Tonalide[®]), Nonylphenol, PFOS, Triclosan, or some steroid estrogens partition by a considerable amount onto sludge (Heidler and Halden, 2008; Jelic et al., 2012).

Water bodies that receive a large amount of WWTP effluents are significantly influenced by the influx of micropollutants that pass through WWTPs. Therefore, the adverse effect of WWTP effluents on the quality of river water is significant and cannot be underestimated. The presence of small concentrations of PPCPs or other micropollutants has been associated to chronic toxicity (Carlsson et al., 2009; Gilbert, 2011; Sanchez et al., 2011; Santos et al., 2010), endocrine disruption (feminization, intersex, and lower sperm counts in wild fish populations) (Baynes et al., 2012 and references therein; Kidd et al., 2007), and the development of antibiotic pathogen resistance (Lubick, 2011). They might also affect water bodies that have an important function as drinking water resources, as well as infiltrate into groundwater (Loos et al., 2010; Stuart et al., 2012), with possible effects on human health.

In 2006, Reemtsma and co-workers (2006) published the first EU-wide study on the occurrence of polar organic pollutants in WWTP effluents and the receiving surface waters. In this study, the effluents of eight municipal WWTPs in Western Europe were analyzed by liquid chromatography - mass spectrometry (LC-MS) for the occurrence of 36 polar pollutants, comprising PPCPs and other household and industrial chemicals.

Moreover, in a long-term study of the effluents of three WWTPs over 10 months Benzotriazoles, Benzothiazole-2-sulfonate, Diclofenac, and Carbamazepine showed mean concentrations of 1-10 micrograms per liter ($\mu\text{g/l}$), followed by some flame retardants, Naphthalene disulfonates, and PPCPs in the range of 0.1-1 $\mu\text{g/l}$. Half of the determined compounds were not significantly removed in tertiary wastewater treatment with enhanced nutrient removal (Reemtsma et al., 2006).

In the last years, several fate studies on the occurrence and behavior of PPCPs, endocrine disruptors, illicit drugs, and other industrial chemicals have been performed. The efficiency of the removal of PPCPs (and other compounds) was found to be strongly dependent on the technology implemented in the WWTP (Hollender et al., 2009; Kasprzyk-Hordern, et al., 2009; Vieno et al., 2007).

In Switzerland, in 2006 the interdisciplinary research project “Micropoll Strategy” was started to develop a strategy for micropollutants removal in municipal wastewater. The aim of the project was to upgrade municipal WWTPs with additional treatment steps to remove micropollutants for the protection of ecosystems and drinking water resources. Complementary treatment steps have been evaluated and it has been shown that water quality can be significantly improved using processes such as powdered activated carbon (PAC) adsorption or ozonation (Micropoll, 2011; 2012).

Currently, tertiary or advanced treatment technologies (e.g. ozonation, chlorine dioxide treatment, UV-Fenton, membrane technologies, Ferrate (VI), sand filters, activated carbon) of secondary effluents are widely discussed as one of the most promising options for the mitigation of micropollutants entering the aqueous environment via discharges from municipal WWTPs (Baynes et al., 2012; Gabet-Giraud et al., 2010; Göbel et al., 2007; Hollender et al., 2009; Joss et al., 2008; Klammer et al., 2010; Klavarioti et al., 2009; Lee et al., 2008, 2009; Nakada et al., 2007; Nowotny et al., 2007; Oller et al., 2011; Radjenović et al., 2007a,b; Stalter et al., 2011). It has already been shown that additional treatment of wastewater reduces endocrine disruption in wild fish (Baynes et al., 2012; Filby et al., 2010; Lee et al., 2008).

The main objective of this research project (“Fate Sees”) was to verify on a European-wide scale the occurrence of as many as possible organic and inorganic chemical contaminants in WWTP effluents, in order to get a European overview.

2. Biological based assays

The chemical-based monitoring of freshwater sources according to the WFD is faced with limitations in the detection of some emerging pollutants as well as in the assessment of the potential effects caused by mixtures of pollutants. Complex mixtures are the rule, not the exception in environmental samples and they can include additive, antagonistic or synergistic interactions of the diverse compounds in a mixture (Kortenkamp et al., 2009). Biological-based analysis has been drawing increasing attention by allowing the evaluation of the effective toxicity of complex environmental samples, often without pre-treatment of the sample or chemical extraction.

Biological-based assays can analyse mixture effects providing data from molecular up to organism level. Special interest is focused on the possibility for fast-screening bioassays using model organisms to provide toxicity and endocrine-disrupting potential of water samples.

The diatom *Thalassiosira pseudonana* and the yeast *Saccharomyces cerevisiae* are both fully sequenced eukaryotic organisms, with recognized applications in ecotoxicology studies. The full genome sequence of the budding yeast *Saccharomyces cerevisiae* has been known since 1996 (Goffeau et al., 1996). Although initially established as a model organism for medical studies, yeast has recently become of high interest also in the ecotoxicology field. An accepted yeast-based bioassay for endocrine disruptors detection has been developed in 1996 (Arnold et al., 1996) and since then, several new bioassays for the detection of hormonal activities have been described (Balsiger et al., 2010; Nguyen et al., 2011). The development of genomics tools in yeast, including yeast disruption and deletion mutants for drug-sensitivity/resistance screening have applications in the identification of targeted genes and pathways for small molecules or therapeutic drugs (Bharucha and Kumar, 2007). Yeast has also been applied to the detection of environmental pollutants-associated molecular pathways, e.g. for cadmium (Serero et al., 2008).

Diatoms are eukaryotic photosynthetic organisms with a worldwide distribution in freshwater and marine environments and they play a dominant role in the global carbon cycle (Smetacek, 1999). Although diatoms have been exploited for decades in environmental monitoring through the use of diatomic indices, the recent full genome sequence of two marine diatoms, the centric *Thalassiosira pseudonana* (Armbrust et al., 2004) and the pennate *Phaeodactylum tricornutum* (Bowler et al., 2008), stirred their potential as model organisms in ecotoxicology. Since then, there has been a steadily increase in research on diatoms, and the diatom *T. pseudonana* has already been the target of various studies based on “omics” approaches to identify cellular changes at the molecular level upon conditions of stress. Thus, changes at the molecular level (transcriptome and/or proteome) have been identified linked with nutrient limitation, temperature and pH (Mock et al., 2008), exposures to copper and hydrogen peroxide (Davis et al., 2006), and to the polycyclic aromatic hydrocarbon benzo(a)pyrene (Carvalho et al., 2011a; Carvalho et al., 2011b; Carvalho and Lettieri, 2011).

The full genome of a freshwater diatom species is yet to be completed, which so far has hindered the application of diatom-based molecular bioassays for monitoring of freshwaters. Despite the fact that *T. pseudonana* has been initially isolated from a coastal zone, *T. pseudonana* cultures have been progressively adapted in the laboratory to lower salinities in order to facilitate toxicity testing of transition and freshwater samples using this marine diatom.

In these studies, 13 effluent water samples were used to set up the screening bioassay based on diatom and yeast growth effects.

In vitro bioassays enable estimation of total biological activity of all compounds that act through the same mode of action present in extracts of any environmental media including waste water. In these studies, presence of toxicants with two important modes of action, i.e. activation of aryl hydrocarbon (AhR) and estrogenic receptor (ER), were investigated in 25 (AhR) and 75 (ER) waste water extracts.

Chemicals that activate aryl hydrocarbon receptor AhR can elicit toxic effects similar to that of 2,3,7,8-Tetrachlorodibenzo-p-dioxin and they are known as dioxin-like chemicals. Related effects include hepatotoxicity, immunotoxicity, dermal toxicity, lethality as well as embryotoxicity, teratogenicity, carcinogenesis or tumor promotion, wasting syndrome and others. Compounds with ability to activate estrogenic receptor ER have the potential to disrupt normal reproduction or developmental processes which are known to lead to adverse health effects such as compromised reproductive capacity, breast and testicular cancer, reproductive dysfunction such as feminization or demasculinization of males and other adverse effects ([Hilscherova et al., 2000](#)). These chemicals cause adverse effects at extremely low concentrations (ng/l) and therefore it is usually difficult and also expensive to detect them by commonly employed instrumental analyses. Reporter gene bioassays utilizing mammalian cells, as used in the present studies, are considered one of the most sensitive tools with respect to detection of estrogens. Evaluation of these toxic potentials also requires high level of expertise ([GWRC, 2006](#)).

3. Description of the campaign and selection of WWTP sampling sites

Annex 1 gives a list of the 91 WWTPs investigated in this study. It contains information on the type of discharges treated in the plant (domestic or industrial), the plant capacity (m^3/day), the capacity in population equivalents, and, if applicable, the type of tertiary treatment applied.

The selection of the WWTPs was done by the participating EU Member States. No selection criteria were given by the JRC. Mainly municipal WWTPs were investigated, but some plants were dominated by industrial waste waters. Unfortunately, not all participants of the campaign (owners of the WWTPs) provided all information requested. Sampling was performed by grab sampling or in many cases also with automated systems as 24h composite samples. One liter water samples, stored in HDPE plastic bottles, were shipped to the JRC by fast courier in thermostated boxes with cooling elements; the samples were stored in a fridge at $\sim 4^\circ\text{C}$, and further distributed as fast as possible to the other expert laboratories. Annex 1 also shows for some samples the storage time (in the fridge), which was in most cases between one and two months.

Wastewater treatment generally comprises a primary, secondary and sometimes an advanced tertiary treatment, employing different physico-chemical and biological techniques. During the primary treatment, solids are removed from wastewater entering for further processing, typically in a biological secondary activated sludge treatment, including nitrification and denitrification, and phosphorus removal.

In some WWTPs, effluent is also disinfected before it is released into the environment (e.g., chlorination, or ultraviolet radiation). In addition, advanced wastewater treatments can be applied to enhance the removal of nitrogen, phosphorus and other pollutants. Tertiary treatment comprises processes such as sand filtration, chlorination, ozonation, or advanced oxidation processes (AOPs) (Radjenović et al., 2007a).

In general, today most WWTPs use activated sludge processes wherein microorganisms are applied to mineralize the pollutants to water and carbon dioxide, or degrade them to acceptable forms. Pollutants can also be removed from water by stripping into air or by sorption onto sludge that is regularly discharged. Some substances may be subject to phototransformation. Therefore, the removal of pharmaceutical residues in activated sludge processes includes four mechanisms: biotransformation, air stripping, sorption and phototransformation (Zhang et al., 2008).

4. Selection of the target compounds

The target compounds analysed in the different laboratories are shown in the following tables. For more information, see also the results and discussions part.

The annual average Environmental Quality Standards (AA-EQS) for inland waters are given for the priority substances (PS) and the newly proposed PS of the WFD.

Table 1: Polar organic chemicals analysed by LC-MS-MS; Lab: JRC-IES, Ispra, Italy. 90 samples analysed; Priority Substances of the WFD in blue.

Pharmaceuticals	Use	Perfluoroalkyl Substances
Diclofenac (EQS: 0.10 µg/l)	Non-steroidal anti-inflammatory drug	PFHxA; perfluorohexanoate
Ketoprofen	Non-steroidal anti-inflammatory drug	PFHpA; perfluoroheptanoate
Naproxen	Non-steroidal anti-inflammatory drug	PFOA; perfluorooctanoate
Ibuprofen	Non-steroidal anti-inflammatory drug	PFNA; perfluorononanoate
Bezafibrate	Drug to lower cholesterol levels	PFDA; perfluorodecanoate
Gemfibrozil	Drug to lower lipid levels	PFBS; perfluorobutansulfonate
Clofibrilic acid	Metabolite of Clofibrate, a lipid lowering drug	PFHxS; perfluorohexansulfonate
Sulfamethoxazole	Antibiotic	PFOS; perfluorooctane sulfonate (EQS: 0.65 ng/l)
Carbamazepine	Anti-convulsion drug to treat epilepsy	
		Pesticides
Personal Care Products		MCPA
N,N'-Diethyltoluamide (DEET)	Insecticide	Mecoprop
Caffeine	Stimulant	2,4-D
Triclosan	Antibacterial substance	Bentazone
		Dichlorprop
Benzotriazoles		2,4,5-T
1H-Benzotriazole	Corrosion inhibitor	Diuron (EQS: 0.2 µg/l)
5-Methyl-1H-benzotriazole	Corrosion inhibitor	Isoproturon (EQS: 0.3 µg/l)
		Atrazine (EQS: 0.6 µg/l)
Sweeteners		Atrazine-desethyl
Sucralose		Simazine (EQS: 1 µg/l)
Acesulfame K		Terbutylazine
		Terbutylazine-desethyl
Nitrophenols		Propazine
1-Nitrophenol		Carbaryl
2,4-Dinitrophenol		Diazinon
		Methabenzthiazuron
Steroid estrogens		Chlortoluron
17β-Estradiol (EQS: 0.4 ng/l)	Natural human estrogen	Hexazinone
Estrone	Metabolite	Linuron
17α-Ethinylestradiol (EQS: 35 pg/l)	Synthetic estrogen (contraceptive)	Metolachlor
		Metoxuron
		Alachlor (EQS: 0.3 µg/l)
		Molinate
		Propanil

Table 2: Pharmaceuticals analysed by LC-MS-MS; Lab: UMEÅ University, Jerker Fick, Sweden;
89 samples analysed; for some selected substances the use is given.

Pharmaceuticals	Use	Pharmaceuticals	Use
Alfuzosin	α_1 -Receptor antagonist to treat benign prostatic hyperplasia	Fluphenazine	
Alprazolam		Flutamid	
Amitryptiline		Glibenclamide	
Atorvastatin		Glimepiride	
Azelastine		Haloperidol	Antipsychotic drug for the treatment of schizophrenia
Biperiden		Hydroxyzine	
Bisoprolol	Beta blocker to treat cardiovascular diseases	Ibersartan	Angiotensin II receptor antagonist for the treatment of hypertension
Bromocriptin		Loperamide	
Buprenorphin		Maprotilin	
Bupropion	Antidepressant and smoking cessation aid	Meclozine	
Chloprothixen		Memantin	
Chlorpromazine		Mianserin	
Cilazapril		Miconazole	
Citaprolam	Antidepressant drug	Nefazodon	
Clemastine		Orphenadrin	Parkinson's Disease drug
Clomipramine		Oxazepam	Treatment of anxiety and insomnia
Clonazepam		Paroxetin	
Clotrimazol		Perphenazine	
Codeine	Analgesic opium alkaloid	Pizotifen	
Cyproheptadine		Promethazin	
Dicycloverin		Ranitidine	
Diltiazem	Calcium channel blocker to treat hypertension, and angina pectoris	Repaglinide	Blood glucose-lowering drugs to treat type II diabetes
Diphenhydramin	Sedative to treat allergies and insomnia	Risperidone	Antipsychotic drug for the treatment of schizophrenia
Duloxetine		Rosuvastatin	
Eprosartan	Angiotensin II receptor antagonist to treat high blood pressure	Sertraline	
Etonogestrel	Progestin used in hormonal contraceptives	Tamoxifen	
Fenofibrate		Telmisartan	Angiotensin II receptor antagonist to treat hypertension
Fentanyl		Terbutalin	
Fexofenadine	Antihistamine drug to treat hayfever, allergy symptoms, and urticaria	Tramadol	Opioid analgesic used in treating severe pain
Flecainide	Antiarrhythmic agent to treat tachyarrhythmias (abnormal fast rhythms of the heart)	Trihexyphenidyl	Antiparkinsonian agent
Fluconazole	Triazine antifungal drug	Venlafaxin	Antidepressant drug
Fluoxetine		Verapamil	
Flupetixol		Zolpidem	

Table 3: Organophosphate ester flame retardants; Lab: UMEÅ University, Peter Haglund, Sweden; 89 samples analysed.

Organophosphate ester flame retardants
Tri-iso-butylphosphate (TIBP)
Tributylphosphate (TBP)
Tris(2-chloroethyl)phosphate (TCEP)
Tris(2-chloroisopropyl)phosphate (TCPP)
Tris(1,3-dichloro-2-propyl)phosphate (TDCP)
Tris(2-butoxyethyl)phosphate (TBEP)
Triphenylphosphate (TPP)
2-Ethylhexyldiphenylphosphate (EHDPP)
Tris(2-ethylhexyl)phosphate (TEHP)
Tricresylphosphate (TCP)

Table 4: X-ray contrast agents and Gadolinium compounds; Lab: IWW, Germany; 73 samples analysed.

X-ray contrasting agents	Gadolinium from magnetic resonance imaging contrast media used in hospitals
Amidotrizoic acid	Gadolinium (Gd)
Iohexol	anthrophogenic Gd
Iomeprol	Gd-Anomaly
Iopamidol	
Iopromid	
Iothalamic acid	

Table 5: Veterinary drugs / antibiotics analysed by LC-MS-MS; Lab: VITO, Belgium; 30 samples analysed.

	Pharmaceutical class	Active substance
ANTIBIOTICS	Tetracyclines	Oxytetracycline
		Doxycycline
	Macrolides	Tylosin
		Tilmicosin
	β -lactam antibiotics - Penicillins	Penicillin V
		Penicillin G (benzylpenicillin)
		Ampicillin
		Amoxicillin
	Sulfonamides	Sulfamethoxazol
		Sulfadoxin
		Sulfadiazin
	Quinolones	Flumequine
		Enrofloxacin
		Ciprofloxacin
	Lincosamides	Clindamycin
		Lincomycin

	Other	Trimethoprim
		Florfenicol
		Tiamulin
ANTI-PARASITIC	Imidazothiazoles	Levamisole

Table 6: Siloxanes and musk fragrances; Lab: UBA, Vienna, Austria; 77 samples analysed.

Siloxanes	Musk fragrances
Decamethylcyclopentasiloxan (D5)	Cashmeran
Decamethyltetrasiloxan (MD2M)	Celestolid® (ADBI)
Dodecamethylcyclohexasiloxan (D6)	Galaxolid® (HHCB)
Dodecamethylpentasiloxan (MD3M)	Phantolid® (AHMI)
Octamethylcyclotetrasiloxan (D4)	Tonalid® (AHTN)
Octamethyltrisiloxan (MDM)	Traesolid® (AITI)

Table 7: Trace elements analysed by ICP-AES; Lab: JRC-IES, Ispra, Italy; 90 samples analysed; MAC-EQS for mercury.

Trace elements using ICP-AES
Hg (EQS: 0.07 µg/l) , Ag, Al, As, Ba, Be, Cd (EQS: 0.08-0.25 µg/l), Co, Cr, Cu
Mg, Mn, Mo, Ni (EQS: 4 µg/l), Pb (EQS: 1.2 µg/l), Sb, Se, Ti, Zn

Table 8: Toxicity analysis by in vitro reporter gene bioassays; Estrogenicity equivalents (EEQ) and Dioxin-like toxicity (TEQbio); Lab: RECETOX, Masaryk University, Brno, Czech Republic; 75 samples (EEQ) and 25 samples (TEQbio) analysed.

Estrogenicity equivalents (EEQ)	Dioxin-like toxicity (TEQbio)
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Table 9: Acute toxicity to yeast and diatom cultures measured by optical bioassays; Lab: JRC-IES, Ispra, Italy; 13 samples analysed.

Yeast culture bioassay (strain W303.1a)	Thalassiosira pseudonana culture bioassay (strain CCMP 1335)
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5. Analytical methods

5.1. Multi-compound SPE-LC-MS-MS method (JRC-IES)

5.1.1. Sample preparation / Solid-phase extraction (SPE)

The water samples were extracted at the JRC by solid-phase extraction (SPE) with Oasis[®] HLB (200 mg) cartridges. Most water samples contained particles (suspended particle material; SPM) which settled to the bottom of the plastic bottles. The water was not filtered, but decanted into a 500 ml glass bottle (Schott-Duran). Thus, only the dissolved (liquid) water phase was investigated. Before extraction, the samples (500 ml) were spiked with the internal standard (50 µl), which contained the labeled substances 2,4-D d3, MCPA d3, PFOA ¹³C₄, PFNA ¹³C₅, PFOS ¹³C₄, Ibuprofen ¹³C₃, Triclosan d10, Estrone d2, Sucralose d6, Carbamazepine d10, Simazine ¹³C₃, Atrazine ¹³C₃. The spiking level in the water samples was 10 ng/l for PFOA ¹³C₄, PFNA ¹³C₅, and PFOS ¹³C₄, one microgram per liter for Sucralose d6, and 100 ng/l for the other labeled compounds. The glass bottles were closed, and then the samples were mixed by shaking for some seconds.

The SPE procedure for the clean-up and concentration of water samples was performed automatically using an AutoTrace[®] SPE workstation (Thermo Scientific). 200 mg (6 ml) Oasis[®] HLB columns (Waters, Milford, MA, USA) were used. The cartridges were activated and conditioned with 5 ml methanol and 5 ml water at a flow-rate of 5 ml/min. The water samples (400 ml) were passed through the wet cartridges at a flow-rate of 5 ml/min, the columns rinsed with 2 ml water (flow 3 ml/min), and the cartridges dried for 30 min using nitrogen at 0.6 bars. Elution was performed with 6 ml methanol. Evaporation of the extracts with nitrogen to 500 µl was performed at a temperature of 35°C in a water bath using a TurboVap[®] II Concentration Workstation (Caliper Life Sciences).

Some selected effluent water samples are shown in Figure 1:



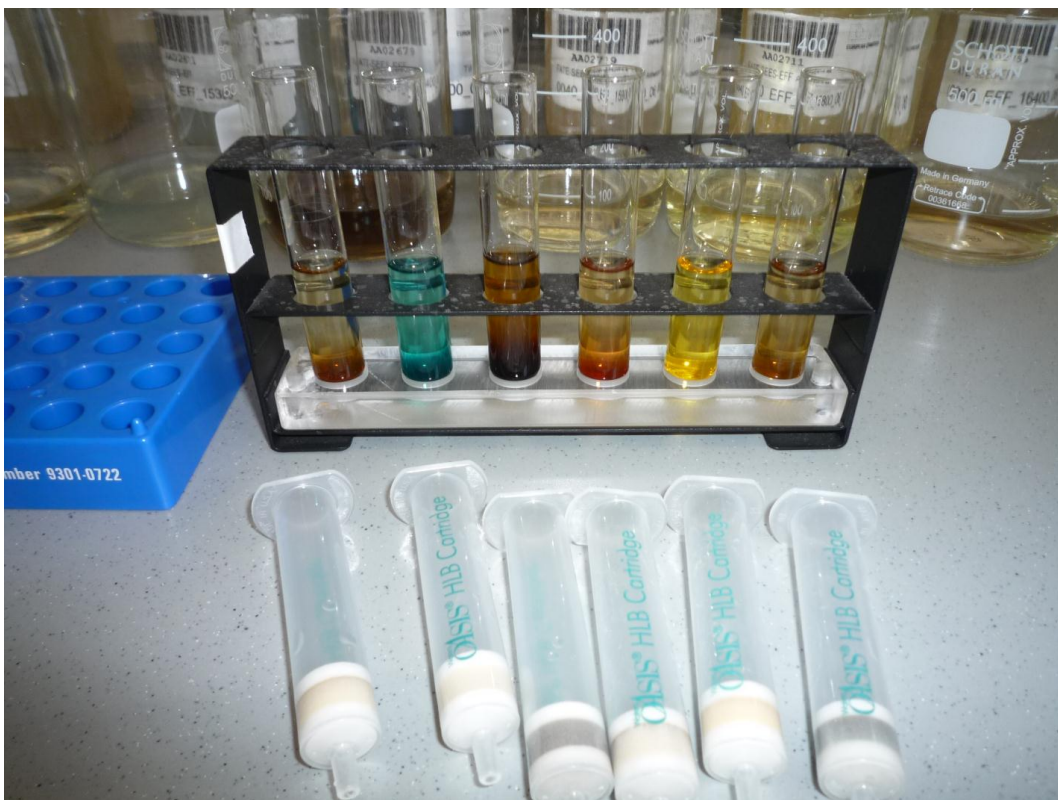


Figure 1: Sampling bottles, water extracts, and SPE cartridges.

5.1.2. Liquid chromatography tandem mass spectrometry (LC-MS²)

Analyses were performed by reversed-phase liquid chromatography (RP-LC) followed by electrospray ionization (ESI) mass spectrometry (MS) detection using atmospheric-pressure ionization with a triple-quadrupole MS-MS system. Quantitative LC-MS² analysis was performed in two separate LC-MS² runs in the multiple reaction monitoring (MRM) mode in negative and positive ionization.

LC was performed with an Agilent 1100 Series LC systems consisting of a binary pump, vacuum degasser, autosampler and a thermostated column compartment. LC separations were performed with a Hypersil Gold column (Thermo Electron Corp., 100 × 2.1 mm, 3 μm particles). Tandem mass spectrometry was performed on a bench-top triple-quadrupole *quattro micro* MS from Waters-Micromass (Manchester, UK) equipped with an electrospray probe and a Z-spray interface.

The eluants used for the separations of the target analytes were water and acetonitrile. The water phase used was acidified with 0.1% acetic acid (pH 3.5) in the negative and positive ionization modes. The flow-rate was 0.25 ml/min. The gradient started with 90% water and proceeded to 90% acetonitrile over 25 min, conditions hold for 5 min, returned back to the starting conditions over 5 min, and followed by 5 min equilibration. The injection volume was 5 μl; injection was performed by the autosampler.

Instrument control, data acquisition and evaluation (integration and quantification) were done with MassLynx software. Nitrogen is used as the nebulizer gas and argon as the collision gas. Capillary voltage was operated at 3.2 kV, extractor lens at 1.0 V, and RF lens at 0.0 V. The source and desolvation temperatures were set to 120 and 350°C under chromatographic HPLC conditions. Cone and desolvation gas flows were 50 and 600 l/h, respectively. The applied analyser parameters for MRM analysis were: LM 1 and HM 1 resolution 11.0, ion energy 1 1.0, entrance -1 (negative mode), 2 (positive mode), exit 1, LM 2 and HM 2 resolution 11.0, ion energy 2 2.0, multiplier 600 V. The MRM inter-channel delay was 0.05 and the inter-scan delay 0.15.

Collision-induced dissociation (CID) was carried out using argon at approx. 3.5×10^{-3} mbar as collision gas at collision energies of 7 - 40 eV. The optimized characteristic MRM precursor → product ion pairs monitored for the quantification of the compounds are given in Table 10.

Table 10: LC-MS² parameters, retention times, recoveries, LOQs (JRC). MRM = multiple reaction monitoring; SPE recovery rates from 400 ml Milli water spiked at 100 ng/l using 200 mg Oasis HLB cartridges; LOQ = method detection limits; internal standards are grey-shaded; priority substances of the WFD in **blue**; Carb. = Carbamazepine; second transition in brackets.

Compound	CAS no.	MRM transitions	Ret.Time [min]	Recovery [%]	LOQ [ng/l]	Internal Standard
Negative mode						
Acesulfame K	55589-62-3	162 > 82	2.4	10 ± 9	10	Sucralose d6
Sucralose	56038-13-2	395 > 359	5.5	62 ± 9	100	Sucralose d6
Sucralose d6		401 > 365	5.5	64 ± 7		Sucralose d6
Nitrophenol	100-02-7	138 > 108	10.4	58 ± 7	5	2,4-D d3
2,4-Dinitrophenol	51-28-5	183 > 109	11.8	63 ± 6	5	2,4-D d3
Bentazone	25057-89-0	239 > 132	13.1	62 ± 8	1	2,4-D d3
2,4-D	94-75-7	219 > 161	14.9	56 ± 8	1	2,4-D d3
2,4-D d3		222 > 164		58 ± 7		
MCPA	94-74-6	199 > 141	15.1	63 ± 8	0.5	MCPA d3
MCPA d3		202 > 144	15.1	63 ± 7		
Ketoprofen	22071-15-4	253 > 209	15.8	71 ± 8	10	MCPA d3
Naproxen	22204-53-1	229 > 169	16.1	65 ± 8	5	2,4-D d3
Bezafibrate	41859-67-0	360 > 274	16.4	66 ± 12	1	MCPA d3
Mecoprop	7085-19-0	213 > 141	16.4	62 ± 8	1	MCPA d3
Dichlorprop	120-36-5	233 > 161	16.4	64 ± 8	3	MCPA d3
Clofibric acid	882-09-7	213 > 127	15.5	65 ± 6	2	MCPA d3
2,4,5-T (Trichlorophenoxyacetic a.)	93-76-5	255 > 197	16.7	71 ± 8	2	MCPA d3
Ibuprofen	15687-27-1	205 > 161	19.1	62 ± 12	2	Ibuprofen ¹³ C ₃
Ibuprofen ¹³ C ₃		208 > 163.4	19.1	65 ± 9		
Diclofenac	15307-86-5	294 > 250	18.8	65 ± 6	1	Ibuprofen ¹³ C ₃
Gemfibrozil	25812-30-0	249 > 121	20.7	62 ± 9	1	Ibuprofen ¹³ C ₃
Triclosan	3380-34-5	287 > 35	21.6	55 ± 7	4	Triclosan ¹³ C ₁₂
Triclosan ¹³ C ₁₂		299 > 35	21.6			
Perfluoroalkyl Substances						
PFHxA; perfluorohexanoate	68259-11-0	313 > 269	17.2	61 ± 12	1	PFOA ¹³ C ₄
PFHpA; perfluoroheptanoate	375-85-9	363 > 319	19.4	81 ± 14	1	PFOA ¹³ C ₄
PFOA; perfluorooctanoate	335-67-1	413 > 369	22.2	85 ± 11	1	PFOA ¹³ C ₄
PFOA ¹³ C ₄		417 > 372	22.2	82 ± 9		
PFNA; perfluorononanoate	375-95-1	463 > 419	25.0	85 ± 9	0.5	PFNA ¹³ C ₅
PFNA ¹³ C ₅		467.5 > 422.7	25.0	85 ± 9		
PFBS; perfluorobutansulfonate		298.8 > 79.7	17.7	59 ± 9	1	PFOS ¹³ C ₄
PFHxS; perfluorohexansulfonate		398.8 > 79.7	24.2	63 ± 8	1	PFOS ¹³ C ₄
PFOS; perfluorooctansulfonate	1763-23-1	499 > 80 (99)	28.3	65 ± 10	0.5	PFOS ¹³ C ₄
PFOS ¹³ C ₄		503 > 80 (99)	28.3	66 ± 12		
PFDA; perfluorodecanoate	335-76-2	513 > 469	27.7	76 ± 8	1	PFNA ¹³ C ₅
Positive mode						
Caffeine	58-08-2	195 > 138	3.2	75 ± 8	5	Carb. d10
1H-Benzotriazole	95-14-7	120.1 > 64.6 (92)	5.0	56 ± 9	40	Carb. d10
5-Methyl-1H-benzotriazole	13351-73-0	134.1 > 78.6 (77)	8.8	47 ± 8	40	Carb. d10
Atrazine-desethyl	6190-65-4	188 > 146	8.2	71 ± 10	1	Simazine ¹³ C ₃
Sulfamethoxazole	723-46-6	254 > 156	9.4	64 ± 9	1	Carb. d10
Metoxuron	19937-59-8	229 > 71.6	11.0	68 ± 5	1	Simazine ¹³ C ₃

Hexazinon	51235-04-2	253 > 171	11.4	70 ± 7	2	Simazine ¹³ C ₃
Terbutylazine-desethyl	30125-63-4	202 > 146	12.5	70 ± 8	1	Simazine ¹³ C ₃
Simazine	122-34-9	202 > 132	11.4	70 ± 11	5	Simazine ¹³ C ₃
Simazine ¹³ C ₃		205 > 134	11.4	72 ± 9		
Carbamazepine	298-46-4	237 > 194	13.1	68 ± 11	1	Carb. d10
Carbamazepine d10		247 > 204	13.1	69 ± 8		
Methabenzthiazuron	18691-97-9	222 > 165	13.5	64 ± 9	3	Simazine ¹³ C ₃
Chlortoluron	15545-48-9	213 > 71.6	13.9	62 ± 8	3	Atrazine ¹³ C ₃
Atrazine	1912-24-9	216 > 174 (132)	13.9	72 ± 13	1	Atrazine ¹³ C ₃
Atrazine ¹³ C ₃		219 > 177	13.9	73 ± 8		
Carbaryl	63-25-2	202 > 145	14.1	55 ± 7	3	Atrazine ¹³ C ₃
N,N'-Diethyltoluamide (DEET)	134-62-3	192 > 119	14.4	75 ± 6	1	Atrazine ¹³ C ₃
Isoproturon	34123-59-6	207 > 72	14.6	72 ± 13	1	Atrazine ¹³ C ₃
Diuron	330-54-1	233 > 72	14.8	72 ± 12	1	Atrazine ¹³ C ₃
Terbutylazine	5915-41-3	230 > 174 (132)	16.6	69 ± 8	1	Atrazine ¹³ C ₃
Linuron	330-55-2	249 > 160	17.1	57 ± 7	5	Atrazine ¹³ C ₃
Metolachlor	51218-45-2	284 > 252	18.8	64 ± 11	3	Atrazine ¹³ C ₃
Alachlor	15972-60-8	270 > 238	19.0	58 ± 8	3	Atrazine ¹³ C ₃
Diazinon	333-41-5	305 > 169	20.7	56 ± 9	1	Atrazine ¹³ C ₃

5.1.3. Identification, quantification, QA/QC and LOQs

The first mass transition in Table 10 was used for quantification, and the second (between brackets) only for confirmation purposes. For most compounds the second transition was not sensitive enough. The internal standards 2,4-D d3, MCPA d3, Ibuprofen ¹³C₃, Atrazine ¹³C₃ and Simazine ¹³C₃ were purchased from Cambridge Isotope Laboratories (Andover, MA, USA), Carbamazepine d10 from CDN ISOTOPES (Quebec, Canada), PFOS, PFOA, and PFNA ¹³C standards from Wellington Laboratories (Guelph, Canada). The recoveries were determined with spike experiments in the concentration range of 10 and 100 ng/l using Milli-Q water (replication n=6); they were in the range of 50-90% (see (Loos et al., 2007)).

The compounds were identified by retention time match and their specific LC-MS² MRM transitions. Good performance of the developed analytical methods was demonstrated by successful participation in several interlaboratory exercises on non-steroidal anti-inflammatory drugs (NSAIDs) (Farré et al., 2008; Heath et al., 2010), perfluoroalkyl substances (PFASs) (van Leeuwen et al., 2009) and nonyl- and octylphenol (Loos et al., 2008b). Quantification of the individual compounds was performed with similar internal standards (IS).

For instance, the first compounds (pesticides, pharmaceuticals) in Table 10 were all quantified with the internal standard ibuprofen ¹³C₃. All perfluorinated carboxylates were quantified with PFOA ¹³C₄, and PFOS with PFOS ¹³C₄. Sulfamethoxazole, Carbamazepine, Caffeine, and the Benzotriazoles were quantified with the IS Carbamazepine d10. The pesticides and metabolites were quantified with Atrazine ¹³C₃ and Simazine ¹³C₃. The relative response factors of the compounds in relation to the IS were calculated in all cases. Thus, the reported concentrations are corrected with the

recoveries of the compounds. A comparative check of internal quantification was always performed with external quantification.

The compound-dependent method detection limits (MDLs or LODs) for the SPE-LC-MS² procedure were calculated from the mean concentrations of the blanks of the real water samples plus 3 times the standard deviation; 400 ml water was extracted and concentrated to 0.5 ml, which results in an enrichment factor of 800.

5.1.4. Measurement uncertainty

Measurement uncertainties of analytical methods can be estimated based on the method performance criteria obtained from method validation, combined with the outcome of the analysis of certified reference materials (CRM), or from the z-scores derived from interlaboratory studies. CRMs for polar organics in water samples do not exist. The JRC-IES laboratory participated in two interlaboratory studies on non-steroidal anti-inflammatory drugs NSAIDs (Farré et al., 2008; Heath et al., 2010), the 3rd international interlaboratory study on PFASs (van Leeuwen et al., 2009), and a dedicated study on Nonyl- and Octylphenol (Loos et al., 2008b). The measurement uncertainty is estimated to be around 25-50%; it was shown that typical uncertainties for the analysis of water samples by SPE-GC-MS are in the range of 25-50% (Planas et al., 2006).

5.1.5. Statistical analyses

Frequency of positive detection (freq) in [%], average, median (med), and percentile 90% (Per90), were quantified with excel software (Microsoft).

5.2. Organophosphate ester flame retardants (UMEA)

5.2.1. Sample preparation and clean-up

The effluent samples (100 ml) were extracted according to Bester (2005) using liquid-liquid extraction with toluene after adding an aliquot of internal standard solution ²⁷D tri-*n*-butylphosphate (²⁷D-TBP). The organic phase was separated from the aqueous one after freezing the samples overnight at -20 °C. The effluent extracts were concentrated to approximately 1 ml by rotary evaporation, quantitatively transferred to conical GC vials, and further reduced to 100 µl under a gentle stream of nitrogen.

5.2.2. Instrumental analysis

The samples were analyzed using a GC-HRMS high-resolution system consisting of an Agilent Technologies 6890 GC equipped with a CTC Analytics autosampler and coupled to a Micromass AutoSpec-Ultima mass spectrometer tuned to a resolution of 8000. 1 µl of each sample was injected into the GC, which was operated in splitless mode (2 mins splitless time). The injector temperature was set to 250°C and gas chromatographic separation was carried out using a DB-5 fused silica capillary column (length 30 m, i.d. 0.25 mm, film thickness 0.25 µm) from J&W Scientific (Folsom, CA). The GC oven was initially held at 80°C for 4 mins, increased to 190°C at a rate of 15°C/mins, and then at

10°C/mins to the final temperature, 310°C, which was maintained for 4 mins. Helium was used as a carrier gas at a flow rate of 1.3 ml/mins. The MS was operated in selected ion monitoring (SIM) mode with electron ionization at 37 eV. The OPs were identified by comparing mass fragment ratios and retention times of sample components and reference standards, and quantified using the internal standard technique, which automatically corrects the data for losses during sample work-up and analysis.

5.2.3. Quality control

The following quality criteria were used to ensure correct identification and quantification of the target compound: (a) the retention time should match those of the standard compounds within ± 0.05 min, (b) the intensity ratios of the selected ions (target- and qualifier-ions) are within $\pm 15\%$ of expected/theoretical value (c) the signal-to-noise ratios are greater than 3:1. An analytical method blank was included for each sample batch analysed to assess background interferences and possible contamination of the samples. The data was blank corrected if the background contributed with less than 10% of the sample concentration. Internal standard was added to the sample at the start of the working-up procedure of the sample. The internal standard has similar chemical and physical properties to the compounds to be analyzed. A recovery rate of $>70\%$ was considered acceptable (but was usually higher).

5.3. Pharmaceuticals (UMEA)

5.3.1. Sample preparation / Solid-phase extraction (SPE)

The water samples were extracted by solid-phase extraction (SPE) with Oasis[®] HLB (200 mg) cartridges (Waters, Milford, MA, USA). Before extraction, the samples (100 ml) were filtered through a 0.45 μm membrane filter (MF, Millipore, Sundbyberg, Sweden) and acidified to pH 3 using sulphuric acid. Five nanograms of each internal surrogate standard were added to each sample. The glass bottles were closed, and then the samples were mixed by shaking.

The cartridges were activated and conditioned with 5 ml methanol and 5 ml water at a flow-rate of 5 ml/min. The water samples were passed through the wet cartridges at a flow-rate of 5 ml/min, the columns rinsed with 2 ml water (flow 3 ml/min), and the cartridges dried for 10 min using air. Elution was performed with 5 ml methanol followed by 3 ml ethylacetate. The eluates were evaporated to 20 μl under a gentle air stream, and dissolved in 5% acetonitrile in water, with 0.1% formic acid, to a final volume of 1.0 ml.

5.3.2. LC-MS-MS analyses

Analyses were performed by reversed-phase liquid chromatography (RP-LC) followed by heated electrospray ionization (HESI), in positive and negative mode, mass spectrometry (MS) detection with a triple-quadrupole MS-MS system.

LC was performed with an Accela LC pump (Thermo Fisher Scientific, San Jose, CA, USA) and a PAL HTC autosampler (CTC Analytics AG, Zwingen, Switzerland).

Tandem mass spectrometry was performed on a bench-top triple-quadrupole TSQ Quantum Ultra EMR (Thermo Fisher Scientific, San Jose, CA, USA).

The eluants used for the separations of the target analytes were water, methanol and acetonitrile. The water phase used was acidified with 0.1% formic acid. The elution conditions were programmed as follows: 200 µl/min of 100% water for 1 min followed by a gradient change to 20/20/60 water/ACN/MeOH at a flow of 250 µl/min in 8 min, and final gradient change to ACN/MeOH 40/60 at a flow of 300 µl/min in 11 min. These parameters were held for 1 min and then changed to starting conditions and retained 4 min to equilibrate the column for the next run. The injection volume was 20 µl; injection was performed by the autosampler. All experiments were performed at 22°C ambient temperature.

Instrument control, data acquisition and evaluation (integration and quantification) were done with Excalibur software (Thermo Fisher Scientific, San Jose, CA, USA). Nitrogen is used as the nebulizer gas and argon as the collision gas. Capillary voltage was operated at 3.5 kV. Heated electrospray (HESI) in positive or negative ion mode was used for ionization. Fused-silica capillary in the spray (standard set up) was replaced with metal capillary. The key parameters were set as follows: ionization voltage 3.5 kV; sheath gas 50, and auxiliary gas, 35 arbitrary units; vaporizer temperature 200 °C; capillary temperature 325°C; and collision gas (argon) flow 1.5 ml/min. Both first and third quadrupoles were operated at a resolution of 0.7 FMWH.

Collision-induced dissociation (CID) was carried out using argon at approx.1.5 mTorr as collision gas at collision energies of 10-59 eV. The optimized characteristic MRM precursor product ion pairs monitored for the quantification of the compounds are given in Table 11.

Table 11: LC-MS² parameters, retention times, recoveries, LOQs (UMEÅ).

Compound	MRM transitions	Ret.Time [min]	Recovery [%]	LOQ [ng/l]	Internal standard
Negative mode					
Clonazepam	313.9>278	7.84	82 ± 11	5	Tramadol
Flutamid	275>202	8.72	87 ± 9	5	Amitryptiline
Glibenclamide	492.1>170	9.57	52 ± 43	5	Oxazepam
Glimepiride	489.2>225	9.27	52 ± 38	5	Oxazepam
Positive mode					
Alfuzosin	390.1>156.2	6.85	76 ± 10	0.1	Tramadol
Alprazolam	309.0>205.1	8.18	68 ± 7	10	Tramadol
Amitryptiline	278.1>233.2	8.8	97 ± 5	5	Amitryptiline
Amitryptiline IS	284.1>191.1	8.8			
Atorvastatin	559.2>250.0	9.04	45 ± 21	50	Amitryptiline
Azelastine	382.1>112.2	8.63	95 ± 14	5	Tramadol
Biperiden	312.1>294.3	8.78	101 ± 4	0.1	Amitryptiline

Bisoprolol	326.1>116.2	7.28	145 ± 6	0.1	Tramadol
Bromocriptin	654.1>301.0	8.66	70 ± 31	5	Tramadol
Buprenorphin	468.2>55.4	7.9	64 ± 16	10	Tramadol
Bupropion	240>131.2	7.21	98 ± 9	0.1	Tramadol
Chloprothixen	316>231	9.41	79 ± 9	10	Amitryptiline
Chlorpromazine	319>214	9.23	74 ± 12	5	Amitryptiline
Cilazapril	418.1>114.2	8.5	139 ± 6	1	Tramadol
Citaprolam	325.1>109.2	7.9	65 ± 14	5	Tramadol
Clemastine	344>180	9.65	75 ± 22	0.5	Oxazepam
Clomipramine	315>242.1	9.33	88 ± 9	0.5	Amitryptiline
Clotrimazol	277>165.1	8.9	82 ± 8	1	Amitryptiline
Codeine	300.1>21.15	4.86	90 ± 9	0.5	Tramadol
Cyproheptadine	288.1>191.1	8.55	102 ± 14	5	Tramadol
Dicycloverin	310.1>109.2	10.1	112 ± 17	5	Oxazepam
Diltiazem	415.1>150.1	8.15	101 ± 14	0.5	Tramadol
Diphenhydramin	256.1>165.1	7.82	85 ± 11	0.05	Tramadol
Duloxetine	298.1>123.5	8.7	114 ± 16	1	Tramadol
Eprosartan	425.1>201.1	7.42	106 ± 13	5	Amitryptiline
Etonogestrel	325.1>91.2	9.62	85 ± 12	0.5	Amitryptiline
Fenofibrate	361>139	10.23	52 ± 33	0.5	Carbamazepin
Fentanyl	337.1>105.2	7.6	82 ± 11	50	Fluoxetine
Fexofenadine	502.2>171.1	8.6	106 ± 8	5	Amitryptiline
Flecainide	415.1>301.1	7.94	98 ± 9	0.1	Tramadol
Fluconazole	307.1>238.1	6.06	132 ± 6	0.5	Trimetoprim
Fluoxetine	310.1>44.3	9	110 ± 7	5	Fluoxetine
Fluoxetine IS	315.1>44.3	9			
Flupetixol	435.1>265	10.1	59 ± 32	5	Oxazepam
Fluphenazine	438.1>143.2	9.9	57 ± 30	10	Oxazepam
Haloperidol	376>123.1	7.81	80 ± 21	0.1	Tramadol
Hydroxyzine	375.1>166.1	8.7	106 ± 4	0.5	Amitryptiline
Ibuprofen	429.2>180.1	8.39	102 ± 5	0.5	Amitryptiline
Loperamide	477.2>210.2	9.23	68 ± 17	0.5	Amitryptiline
Maprotilin	278.1>219.2	8.8	97 ± 6	5	Amitryptiline
Meclozine	391.1>200.1	10.03	48 ± 41	5	Oxazepam
Memantin	180.1>107.2	7.87	119 ± 8	0.5	Tramadol
Mianserin	265>118.2	7.9	95 ± 12	1	Tramadol
Miconazole	414.9>159	10.7	60 ± 31	5	Tramadol
Nefazodon	470.1>246.2	9.06	72 ± 17	0.5	Amitryptiline
Orphenadrin	270.1>165.1	8.35	94 ± 6	0.1	Amitryptiline
Oxazepam	287>241.1	7.98	104 ± 13	5	Oxazepam
Oxazepam IS	292>246.1	7.98			
Paroxetine	330>192.1	8.6	87 ± 8	10	Amitryptiline
Perphenazine	404.1>143.2	9.6	71 ± 30	10	Oxazepam
Pizotifen	296>199.1	8.6	92 ± 5	0.5	Amitryptiline

Promethazin	285.1>86.3	8.4	81 ± 6	10	Amitriptyline
Promethazin IS	292.1>89.3	8.4			
Ranitidine	315>176.1	4.62	58 ± 21	0.5	Amitriptyline
Repaglinide	453.2>162.2	8.76	104 ± 3	0.5	Amitriptyline
Risperidone	411.1>110.2	7.2	96 ± 3	0.1	Risperidone
Risperidone IS	415.1>195.1	7.2			
Rosuvastatin	482.1>258.1	8.21	102 ± 12	10	Tramadol
Sertraline	306>19	9.55	84 ± 13	10	Amitriptyline
Tamoxifen	372.2>129.1	10.9	122 ± 10	5	Tamoxifen
Tamoxifen IS	375.2>75.2	10.9			
Telmisartan	515.2>26.17	9.13	79 ± 18	50	Amitriptyline
Terbutalin	226.1>107.2	4.37	27 ± 10	0.5	Tramadol
Tramadol	264.1>246.2	6.37	101 ± 11	0.5	Tramadol
Tramadol IS	268.1>58.4	6.37			
Trihexyphenidyl	302.2>70.3	8.7	97 ± 6	0.1	Amitriptyline
Venlafaxin	278.1>121.3	7.4	101 ± 7	0.5	Tramadol
Verapamil	455.2>165.1	8.26	116 ± 13	10	Tramadol
Zolpidem	308.1>235.2	6.89	103 ± 7	0.5	Tramadol

5.3.3. Identification, quantification, QA/QC and LOQs

The first mass transition in Table 11 was used for quantification, and the second only for confirmation purposes. The internal standards used were: $^2\text{H}_6$ -amitriptyline, $^2\text{H}_5$ -fluoxetine and $^{13}\text{C}_2\text{H}_3$ -tramadol (Cambridge Isotope Laboratories, Andover, MA, USA), $^2\text{H}_5$ -oxazepam, $^2\text{H}_7$ -promethazine, $^2\text{H}_4$ -risperidone, and $^{13}\text{C}_2^{15}\text{N}$ -tamoxifen (Sigma-Aldrich, Steinheim, Germany); which surrogate standards used for which analyte is presented in Table 11.

Absolute recoveries were determined with spike experiments in the concentration range of 1000 and 2500 ng/l using sewage effluent (replication $n = 20$); they were in the range of 27-145% (average 88%) (Table 11).

The compounds were identified by retention time match and their specific LC-MS² MRM transitions. The relative response factors of the compounds in relation to the IS were calculated in all cases. Thus, the reported concentrations are corrected with the recoveries of the compounds. A comparative check of internal quantification was always performed with external quantification.

The compound-dependent limits of quantifications (LOQs) for the SPE-LC-MS² procedure were calculated based on the second point in the calibration curves. 100 ml water was extracted and concentrated to 1 ml, which results in an enrichment factor of 100.

5.4. Antibiotics (VITO)

5.4.1. Compound selection

This study on the occurrence of veterinary active substances in the aquatic environment focused on those pharmaceuticals that are administered to cattle, pigs, horses and poultry. The use of many of these pharmaceuticals are not restricted to veterinary applications but are also used in human medicine. Data on use in Belgium indicate that anti-infectious and anti-parasitic active substances are the most applied. These groups of substances include a variety of different chemical classes, which necessitated a further selection of compounds based on frequency of use. Table 5 (section 3) gives an overview of the compounds of interest that were included in this study.

5.4.2. Chemicals and reagents

High purity (> 95%) analytical standards of the selected pharmaceuticals were purchased from Sigma-Aldrich (Sigma-Aldrich Laborchemikalien GmbH, Seelze, Germany). The individual stock standard solutions were prepared in methanol (MeOH) at a concentration of 500 µg/ml. The working standard solutions in a concentration range from 1 µg/l to 1000 µg/l were prepared by dilution with MilliQ water. All solutions were preserved at 4°C. Certain active substances like quinolones are light sensitive. Therefore, all solutions were prepared in amber bottles.

5.4.3. Sample pre-treatment

The analytical method was optimised in order to cope with the diverse physico-chemical properties of the analytes of interest. Therefore, extractions of sample aliquot were performed at either pH 3 or pH 6. Tetracyclines and sulfonamides tend to form stable complexes with divalent metallic ions like calcium and magnesium. To prevent this complexation, the chelating agent Na₂EDTA·2H₂O was added (Díaz-Cruz and Barceló, 2006; Gros et al., 2006). For a limited set of analytes that were determined at pH 6 the chelating agent was not used. Table 12 shows the various method parameters.

Each pH-adjusted extraction was performed on 250 ml of sample using Oasis HLB SPE cartridges (Waters, Milford, MA, USA). Sample pretreatment was performed as follows. For each effluent sample, 1 aliquot was acidified to pH 3 and 2 aliquots to pH 6 using 6 N HCl. After addition of 250 mg of Na₂EDTA·2H₂O the solution was shaken and stored for 2h. For extractions at pH 3, the Oasis[®] HLB SPE cartridges were conditioned with consecutively 20 ml of MeOH, 6 ml of MilliQ water and 6 ml of acidified water (pH 2). For the extraction of the effluent samples at pH 6 with and without addition of Na₂EDTA·2H₂O, the cartridges were conditioned with 20 ml of MeOH and 12 ml of Milli-Q water.

The effluent sample was loaded on the SPE cartridge, which was washed with 10 ml of MilliQ water to remove residual EDTA and was consecutively dried under reduced atmospheric pressure. The compounds of interest were eluted with 20 ml of MeOH. The extract was evaporated close to dryness and brought to a final volume of 0.5 ml using MeOH.

The optimal conditions for the extraction of the pharmaceutical compounds were determined by the analysis of spiked mineral water. The fortification level of the compounds of interest was 250 ng/l. The recovery was calculated as the ratio between the experimentally obtained concentration and the nominal concentration. For the majority of the compounds (65%), the extraction at acidic conditions (pH 3) resulted in the highest recoveries.

By applying the extraction conditions as listed in Table 12, a recovery above 80% was obtained for the majority of the compounds. On the contrary, amoxicillin and ampicillin are determined with a slightly lower recovery. The recovery of these β -lactam antibiotics varied between 40% and 50%.

Table 12: Extraction parameters of targeted analytes.

pH	EDTA	Analyte
3	Yes	Amoxicillin, ampicillin, penicillin G, penicillin V, sulfadiazine, sulfamethoxazole, sulfadoxin, trimethoprim, oxytetracycline, doxycycline, flumequine, tylosin, tiamulin
6	Yes	Levamisole, ciprofloxacin, enrofloxacin
6	No	Lincomycin, clindamycin, florfenicol, tilmicin

5.4.4. LC-MS-MS analysis

The instrumental analysis was performed using a Waters Acquity UPLC system consisting of an Acquity binary solvent manager, an Acquity sample manager and an Acquity column heater manager. The compounds of interest were separated on an Acquity BEH C₁₈ column (2.1 mm \times 150 mm, 1.7 μ m). The column temperature was kept at 40°C. A gradient elution programme with water: formic acid (0.1%) (solvent A) and acetonitrile: formic acid (0.1%) (solvent B) was used. Chromatographic details are given in Table 13. The flow rate of the mobile phase was 0.4 ml/min. An aliquot of 10 μ l of the final extract was injected into the LC system.

Table 13: Gradient elution programme for the LC-MS-MS analysis of antibiotic substances.

Time (min)	Solvent A (%)	Solvent B (%)
Initial	95	5
0.30	95	5
7.00	5	95
8.00	5	95
8.30	95	5

The UPLC system was coupled to a triple quadrupole MS detector (Micromass Quattro Premier XE, Waters), which was operated in positive electrospray ionisation mode (ESI+). A capillary voltage of 3 kV was used for all compounds. For each compound the cone voltage and the collision energy were optimized, the mass spectrum was recorded and the most abundant ions in the spectrum were subjected to further MSⁿ fragmentations. Out of these results, characteristic precursor and product ions were selected for detection in the multiple reaction monitoring (MRM) mode. These

characteristic precursor and product ions, with the corresponding collision energy (eV) and cone voltage (V) are listed in Table 14.

Table 14: Characteristic precursor and product ions of the selected pharmaceutical compounds.

Compound	Precursor ion	Product ion	Collision energy (eV)	Cone voltage (V)
Oxytetracycline	461	426	23	20
Doxycycline	445	428	23	23
Tylosin	917	174	56	41
Tilmicosin	870	174	55	40
Penicillin V	383	160	26	11
Penicillin G	367	160	23	26
Ampicillin	350	106	17	20
Amoxicillin	349	114	20	20
Sulfamethoxazol	254	92	26	29
Sulfadoxin	311	156	35	17
Sulfadiazin	251	156	17	14
Flumequine	262	244	26	20
Enrofloxacin	360	316	32	17
Ciprofloxacin	332	231	29	35
Clindamycin	427	126	26	29
Lincomycin	407	126	35	29
Trimethoprim	291	230	38	23
Florfenicol	377	243	17	20
Tiamulin	495	192	14	20
Levamisole	205	178	11	20

5.4.5. Method performance

The linearity of the method was determined for each veterinary active substance by injecting standard solutions in the concentration range 1-1000 µg/l. Based on the concentration in the standard solution and the area of the chromatographic peak, the response factor was calculated as the peak area divided by the concentration. Within the working range, the calibration curve fulfilled the requirements for a linear fit. For the selected concentration range, calibration curves were constructed and the squared regression coefficients (R^2) were calculated (Table 15). These regression coefficients were higher than 0.99 for all compounds.

Table 15: Concentration range and corresponding linear regression coefficient (R^2) for the selected veterinary active substances.

Compound	Concentration range	R^2
Oxytetracycline	1-1200 µg/l	0.99972
Doxycycline	1-1100 µg/l	0.99994
Tylosin	60-1300 µg/l	0.99760
Tilmicosin	15-1100 µg/l	0.99940
Penicillin V	1-1100 µg/l	0.99167
Penicillin G	5-1100 µg/l	0.99902
Ampicillin	5-1100 µg/l	0.99988
Amoxicillin	1-1100 µg/l	0.99990
Sulfamethoxazol	6-1200 µg/l	0.99752
Sulfadoxin	5-1100 µg/l	0.99781
Sulfadiazin	1-1100 µg/l	0.99944
Flumequine	8-1700 µg/l	0.99552
Enrofloxacin	50-1000 µg/l	0.99951
Ciprofloxacin	65-1400 µg/l	0.99157
Clindamycin	14-1100 µg/l	0.99967
Lincomycin	1-600 µg/l	0.99658
Trimethoprim	1-1000 µg/l	0.99922
Florfenicol	1-120 µg/l	0.99918
Tiamulin	6-140 µg/l	0.99525
Levamisole	2-145 µg/l	0.99668

Performance characteristics for the analysis in effluents were determined by the duplicate analysis of five samples. The samples were fortified with the pharmaceutical compounds at two concentration levels, i.e. 50 and 250 ng/l. A significant reduction of the recoveries of the pharmaceutical compounds was observed due to matrix suppression (Table 16). This reduction amounted to 20-30% for most compounds. Furthermore, for tilmicosin and tylosin the recovery decreased by more than 50%. Thus matrix effects play an important role in the determination of pharmaceutical compounds in water and suitable countermeasures are necessary to ascertain valid and qualitative results.

As can be seen from the data in Table 15, the analysis of the selected veterinary active substances is characterised by a good precision, despite sometimes a low recovery. With a few exceptions, the relative standard deviation (% RSD) was lower than 15%.

The limit of detection (LOD) and limit of quantification (LOQ) were calculated as three times and six times the standard deviation S_R of the duplicate analyses. These values were compared with the LOQ values determined as concentrations that would give a signal-to-noise ratio of 6 from the chromatograms of both the surface water samples (spiked at low concentration level) and a standard solution (1-2 µg/l). Of these two approaches, the highest LOQ was used for reporting. The resulting LOQ values are listed in Table 16 for the different pharmaceutical compounds. All LOQ values are based on the standard deviation of the duplicate analysis of five surface water samples, with exception of those from amoxicillin, florfenicol and tilmicosin. The LOQ value of these latter

compounds was higher due to the observed signal-to-noise ratio in either the surface water samples or the standard solution.

Table 16: Recovery, reproducibility and limit of quantification (LOQ) of the selected pharmaceuticals in surface water, using the optimal extraction conditions.

Compound	Recovery, % (RSD)		LOQ (ng/l)
	Spiking level 50 ng/l	Spiking level 250 ng/l	
pH 3 with addition of EDTA			
Amoxicillin	6 (58)	5 (52)	25
Ampicillin	19 (18)	7 (46)	25
Penicillin G	85 (13)	104 (8)	50
Penicillin V	75 (21)	85 (12)	50
Sulfadiazin	62 (15)	73 (12)	30
Sulfadoxin	79 (14)	98 (7)	50
Sulfamethoxazole	42 (12)	49 (7)	20
Trimethoprim	66 (5)	75 (5)	10
Oxytetracycline	22 (41)	41 (10)	30
Doxycycline	25 (51)	50 (10)	50
Flumequine	96 (4)	117 (4)	20
Tylosin	122 (11)	103 (11)	50
Tiamuline	49 (7)	44 (17)	20
pH 6 with addition of EDTA			
Levamisole	35 (13)	69 (6)	15
Ciprofloxacin	40 (8)	33 (7)	15
Enrofloxacin	58 (9)	73 (5)	20
pH 6 without addition of EDTA			
Lincomycine	88 (4)	76 (19)	15
Clindamycine	76 (1)	69 (3)	10
Florfenicol	< 10%*	< 10%*	100
Tilmicosin	39 (4)	22 (6)	50

* % RSD values are not mentioned. The standard deviation S_R is comparable with the values of the other compounds. However, due to the very low recovery, very high % RSD values are obtained.

Due to the occurrence of matrix suppression that leads to a strong reduction of recovery the standard addition approach was used. Standard addition means a procedure in which the test sample is divided in three (or more) test portions. One portion is analysed as such, and known amounts of the compound of interest are added to the other test portions. The unknown concentration of the analyte in the sample is derived by extrapolation. Thus a linear response in the appropriate concentration range is essential for achieving accurate results.

Applying the standard addition approach, the MeOH extract (20 ml) was divided into 4 equivalent test portions. One portion was used as such, while the other 3 portions were spiked with an increasing concentration of the compounds of interest: 25 µg/l, 62.5 µg/l and 150 µg/l. Consecutively, the 4 test portions of each sample were concentrated to 0.5 ml, followed by LC-MS-MS analysis.

5.5. X-ray contrast agents (IWW Water Centre)

5.5.1. Reagents, standard substances and stock solutions

Acetonitrile (MS-grade) purchased from LGC-Standards (Wesel, Germany) and water (MS-grade) purchased from Biosolve (the Netherlands) were used as mobile phase for high performance liquid chromatography (HPLC). Water and methanol (both residual grade) used for SPE conditioning, extraction and elution were from LGC Standards (Wesel, Germany). Stock solutions containing 6 x-ray contrast agents of interest dissolved in a mixture of water/methanol (95/5) at six concentration levels (0.01 µg/mL to 0.40 µg/mL) were used as reference solutions. The following x-ray contrast agents listed with their chemical purity (in brackets) were purchased from different suppliers: amidotrizoic acid (97.1%) from Sigma Aldrich (Germany), iohexol (> 95%), iopromide (97.9%) and iothalamic acid (> 95%) from U.S. Pharmacopeia (USA), iomeprol (> 95%) from Bracco (Germany) and iopamidol (97.5%) from Dr. Ehrenstorfer GmbH (Germany). Labeled x-ray contrast agents of high purity were used as internal standards. Amidotrizoic acid-d6 (96%, isotopic purity: 98.5%) was purchased from LGC-Standards (Wesel, Germany) and iopromide-d3 (98%, isotopic purity: 98%) from Campro Scientific (Germany). All stock solutions of x-ray contrast agents with different concentration levels were prepared by volumetric dilution using appropriate solvents and were stored at 4°C in the refrigerator. For spiking of samples these solutions were prepared in water/methanol (95/5).

5.5.2. Sample preparation using solid-phase extraction (SPE)

Non filtered samples (1 liter each) were acidified at pH 3 using HCl (12.5%). Prior to extraction, samples were spiked with labeled internal standard substances (100 µl) to a spiking level of 5 ng/l (amidotrizoic acid-d6) and 20 ng/l (iopromide-d3) respectively. Sample processing was done by a manually operated SPE vacuum device “BAKER spe-10” (Mallinckrodt Baker, Griesheim, Germany) using 3-ml SPE-cartridges filled with 200 mg embedded reversed phase Isolute ENV+ adsorbent (Biotage, Sweden). The cartridges were activated and conditioned with 2 × 3 ml methanol/acetonitrile (50/50) and 2 × 3 ml blank water (pH 3) ensuring that the adsorbent does not run dry. Water samples were passed through the wet cartridges at a flow-rate of 5 ml/min, thereafter the columns were rinsed with 2 × 3 ml water (pH 7), and the cartridges dried for 60 min under reduced pressure using an air stream. Elution was performed with 5 × 2 ml methanol/acetonitrile (50/50). Concentration of extract was performed with a TurboVap® LV Evaporation System from Caliper Life Sciences (Hopkinton, MA, USA) using a gentle stream of nitrogen. The temperature of the eluate during concentration was kept at 40°C by using a water bath. The final solvent volume was adjusted to 1 ml methanol/water (5/95). An aliquot was used for the LC-MS determination.

5.5.3. HPLC-MS-MS

HPLC analyses were performed by using a reversed-phase liquid chromatography (LC) column of the type Gemini C18 (150 mm × 2 mm, 3 µm) from Phenomenex (Torrance, CA, USA) followed by positive electrospray ionization (ESI) mass spectrometry (MS) detection using atmospheric-pressure ionization (API) with a triple-quadrupole tandem mass spectrometric system (Waters TQD-MS-MS, Milford, USA). Quantitative LC-MS/MS analysis was performed in the multiple reaction monitoring (MRM) mode. The m/z values of the precursor ions, product ions, and the collision-induced dissociation (CID) energy for all used transitions within the quantification process are listed in Table 17.

Table 17: X-ray contrast agents. Precursor ions and product ions used for identification and quantification of listed parameters beside further information on mass spectrometric detection.

Compound	CAS no.	Precursor ion (m/z)	Product ion 1 (quantifier) m/z	Product ion 2 (qualifier) m/z	Collision energy eV	Cone voltage V
Amidotrizoic acid	117-96-4	614.69	360.74	232.94	38	42
Iohexol	66108-95-0	821.85	626.50	448.37	40	40
Iomeprol	78649-41-9	777.84	404.80	287.82	59	42
Iopamidol	60166-93-0	777.80	558.64	386.78	40	42
Iopromide	73334-07-3	791.86	572.59	299.69	60	48
Iothalamic acid	2276-90-6	644.88	428.89	176.88	47	38
Amidotrizoic acid-d6	NA	620.73	366.80	239.00	45	36
Iopromide-d3	NA	795.11	575.95	299.94	68	56

5.6. Analysis of Gadolinium anomaly as a measure of Gd-based contrasting agents (IWW Water Centre)

Analyses of Gd and 20 isotopes of other rare earth elements (REE) were carried out by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) with quantitation limits of about 1 ng/l for each of the REE.

The procedure to quantify anthropogenic Gd is the calculation of a so-called Gd anomaly. This procedure is based on the (linear or polynomic) interpolation of the expected Gd concentration from the neighbouring REE after shale normalisation. The Gd anomaly is calculated as the ratio of the measured Gd (after normalisation) to the expected (geogenic) Gd (after normalisation). This approach has been described in the geochemical literature (e.g. by Bau & Dulski, 1996; Möller et al., 2003). It is based on the observation that the REE distribution of geologic materials and of water (that is not anthropogenically influenced) follows a consistent pattern similar to a linear or exponential function if shale-normalised concentrations are plotted against the atomic number of the elements. By using mathematical interpolation methods, the expected value of the (geogenic) normalised Gd can be calculated from the normalized results of the neighbouring REE, and the ratio of measured to expected normalised Gd can be calculated (the general approach is visualised Figure 2. Post-Archaeon Australian shale (PAAS) was used for normalisation (McLennan, 1989).

The fraction of anthropogenic Gd is calculated from the Gd anomaly and the measured Gd concentration as given in Equation (1).

$$Gd_{anthropogenic} [ng / l] = \frac{(Gd_{anomaly} - 1)}{Gd_{anomaly}} * Gd_{measured} [ng / l] \quad (1)$$

This can also be converted to a percentage of the total Gd measured.

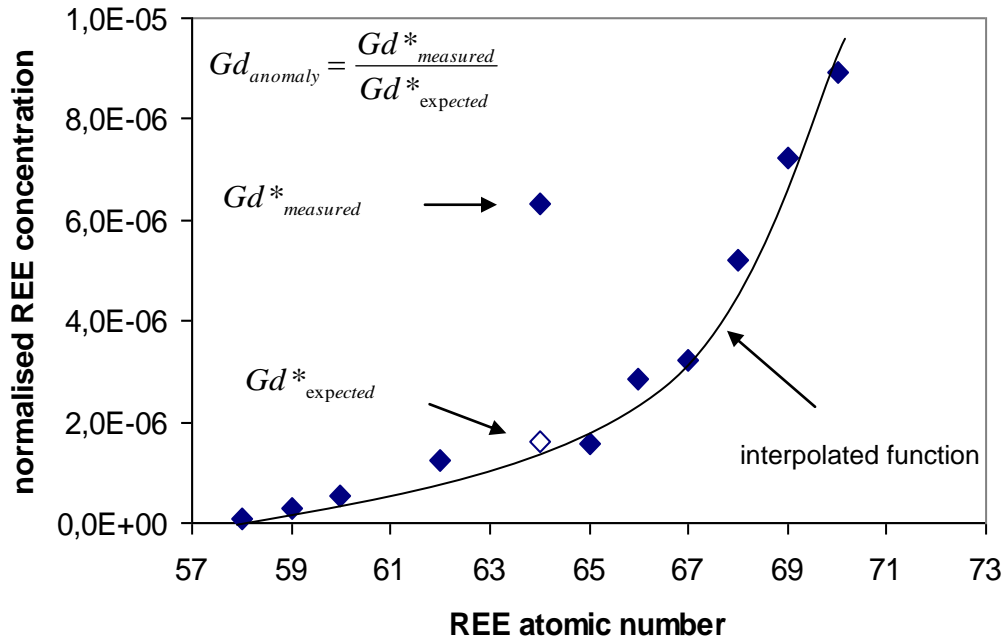


Figure 2: Calculating a Gd anomaly from shell-normalised REE data (from Schwesig & Bergmann, 2011).

5.7. Biological methods (RECETOX)

5.7.1. Sample preparation / Solid-phase extraction (SPE)

Similarly to the chemical methods the water samples were extracted by solid-phase extraction (SPE) with Oasis[®] HLB cartridges with only a few differences. A half liter of each water sample with suspended particle material was manually shaken to simulate turbulent conditions of waste water effluent and filtered through glass fiber filter (2 µm, Fisher Scientific, CZ). Consequently, the samples were applied to the Oasis[®] HLB SPE columns (500 mg, 6 ml). Each column was activated by 6 ml of MeOH and equilibrated by 8 ml of distilled water without applying vacuum. Maximal pressure used for draining the samples was 0.3 bars and average flow rate approximately 5 ml/min. When the

samples passed through the solid phase, the columns were left to dry for about 10 min, and consequently were gravimetrically eluted by 6 ml of MeOH without use of any pressure. Finally, the eluates were concentrated under a gentle nitrogen stream at laboratory temperature to final volumes which corresponded to 1200-times concentrated original effluents. This aliquot was chosen as a maximum concentration which was mostly not cytotoxic to the cells in our previous studies, and enabled detecting estrogenic activity with limits of detection for estrogenicity 0.5 ng/l EEQ (estrogen equivalents) or dioxin-like activity 0.1 ng/l TEQ_{bio} (dioxin toxic equivalents determined with the biotest). Sample extracts were stored at -18°C until analyses.

5.7.2. In vitro bioassays

Two individual bioassays were used to determine estrogenic and dioxin-like potentials of the water extracts. The results were expressed as estrogenic (EEQ) or dioxin-like equivalents (TEQ_{bio}) with respect to standard estrogen (17 β -estradiol (E₂)), or dioxin (2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD)).

To determine estrogenicity of the sample extracts, MVLN (human breast carcinoma) cells stably transfected with luciferase gene under the control of estrogen receptor were used (Novak et al., 2009). Cells were grown in DMEM-F12 without phenol red (Sigma Aldrich, USA) containing 10% fetal calf serum at 5% CO₂ and 37°C. Once the cells reached about 80% confluence they were trypsinized and seeded into a sterile 96-well plate at a density of 25.000 cells/well. For experiments, cells were grown in medium containing foetal calf serum treated with dextran-coated charcoal (strongly reduces concentrations of natural steroids in the serum). After 24 h, the cells were exposed to the dilution series of the tested samples (at least 5 different concentrations of each sample were tested), to the calibration of the reference estrogen - 17 β -estradiol (dilution series 1–500 pM E₂) and blank and solvent controls (0.5% v/v methanol). Exposures were conducted in three replicates for 24 h at 37°C. After the exposure, intensity of the luminescence was measured using Promega Steady Glo Kit (Promega, Mannheim, Germany).

The H4IIE-luc, rat hepato-carcinoma cells stably transfected with the luciferase gene under control of the Arylhydrocarbon receptor (AhR) were used for analysis of dioxin-like activity of the samples (Novak et al. 2009). The used procedure was similar to MVLN bioassay method described above with few differences: H4IIE-luc were plated in DMEM-F12 with phenol red (Sigma Aldrich, USA) containing 10% foetal calf serum with a cell density of 15.000 per well.

5.7.3. Estrogenic and dioxin-like potential quantification

After subtraction of the solvent control response, detected induction of luminescence was related to the maximal response of standard ligand (E₂max for estrogenicity, TCDDmax for dioxin-like compounds) and converted into percentages of E₂max or TCDDmax respectively. The maximal induction as well as the shape of the curve differed among

samples, thus equal efficacy or parallelism of the dose-response curves could not be assumed (Villeneuve et al., 2000). We have used a EEQ₂₅/TEQ_{bio25} estimate (based on the 25% E₂/TCDD_{max} response) to avoid any predictions beyond the measured responses with all samples. EEQ/TEQ₂₅ values were based on relating the amount of E₂/TCDD causing 25% of the E₂/TCDD_{max} response (EC₂₅) to the amount of sample causing the same level of response. Values were determined from the nonlinear logarithmic regression of dose-response curve of calibration standard and samples in Graph Pad Prism (GraphPad Software, San Diego, USA).

5.8. Biological based assays (JRC-IES)

5.8.1. Effluent sample preparation

To perform exposure tests of yeast and diatom cultures to undiluted samples (100% exposure), the effluents were filtrated using a 0.2 µm filter and concentrated to half the volume on falcon tubes by spin vacuum centrifugation at 4°C. Upon exposure, the concentrated effluent was diluted 1:1 (yeast, diatom) and 1:2 (yeast) using concentrated media. This procedure assured that no growth limitation occurred due to lack of nutrient availability in the effluent-exposed cultures as compared to the untreated control yeast cultures.

A total of 13 effluent water samples of WWTPs were tested (Table 18).

Table 18: Samples tested by yeast and diatom assays.

Sample code	Country	Location
#148	Czech Republic	Veslarska
#150	Belgium	TWZ Evergem (2406)
#164	Belgium	WWTP Ronse
#165	Belgium	WWTP Waregem
#166	Belgium	WWTP Deurne
#167	Belgium	WWTP Hasselt
#168	Belgium	WWTP Geel
#195	Ireland	Dublin
#196	Ireland	Oberstown
#202	Austria	AWV Hall i. Tirol-Fritzens
#209	Spain	Ulldecona
#210	Spain	Godall
#257	NL	WWTP Leek (Noorderzijlvest)

5.8.2. Yeast culture

The yeast strain W303.1a was selected for the test because of its increased sensitivity to oxidative stress. W303.1a is a haploid strain with genotype MATa (*leu2-3,112 trp1-1 can1-100 ura3-1 ade2-1 his3-11,15*), carrier of a *ybp1-1* mutation that increase peroxide sensitivity (Veal et al., 2003). A small scale bioassay performed in broth (Hasenbrink et al., 2006) was optimized and adapted to a 96-well microtiter plate for fast-screening of multiple samples. The culture was grown in minimal media (YNB, Sigma) to prevent any interference from protein binding to chemicals, with addition of limiting nutrients (Tryptophan, Uracil, Adenine, Histidine and Leucine) for 48 hours before exposure. The cell cultures were added to each test mix (effluent in culture media) and to the control (just media) at the desired starting cell density (corresponding to an $\text{Abs}_{600\text{ nm}} \approx 0.3$) as measured on a spectrophotometer. The cultures were then distributed, in triplicates of 200 μl , in a 96-well plate (Sarstedt). The measured cell densities were corrected by removing the optical density contribution of each effluent diluted in the media at the same concentration as in the cultures.

The optical density was measured immediately after addition of the cells to the effluent mixture (time zero), and after 2, 4, 6, 8, 10, 24, 30 and 48h of exposure.

Three replicates were performed using the same starting culture for all control and effluent exposure conditions in each experiment replicate.

5.8.3. Diatom culture

T. pseudonana (strain CCMP 1335) was obtained as axenic culture from the Provasoli-Guillard National Center for Culture of Marine Phytoplankton (CCMP, West Boothbay Harbour, Maine, USA). Diatoms were maintained at around 14°C under a diurnal light cycle of 13 h light and 11 h darkness. The culture medium was f/2-medium, based on artificial sea water with a salt concentration of 32 g/l Sodiumchloride (NaCl) (ASW, Sigma-Aldrich, Steinheim, Germany). *T. pseudonana* was cultured at densities between 0.75×10^6 and 2×10^6 cells/ml. Fresh cultures for maintenance were inoculated every 7 days. Cell densities were determined by measuring the absorption at 450 nm using a microplate spectrophotometer (Biorad, Hercules, CA, USA) and used to calculate growth rates, as previously described (Bopp and Lettieri, 2007). *T. pseudonana* cultures were progressively adapted to decreasing salinities weekly, i.e. 24, 16 and 8 g/l, and let grown at the final salinity for at least one month prior to the tests.

The culture protocol was adapted to a 96-well plate and exposure to the effluents (at the original concentration) was followed by measuring the cell density immediately after addition of the effluent (time zero), and after 24, 48 and 72h of exposure. Prolonged shaking of the plate was required (at least 20s) to re-suspend the cells prior to the measurements.

For those effluents eliciting growth inhibition, a serial dilution of the effluent was performed and exposed to the diatom cultures at the different salinities, to find the lowest dilution eliciting the harmful effect.

6. Results and discussion

6.1. SPE-LC-MS² analysis of the target compounds (JRC-IES)

The SPE-LC-MS² procedure for the analysis of selected polar organic chemicals was developed and optimized before (Loos et al., 2007, 2008, 2009). Figure 3 shows an LC-MS² chromatogram of a standard solution in the negative ionization mode, and Figure 4 in the positive ionization mode. In the negative mode, three detection windows were used.

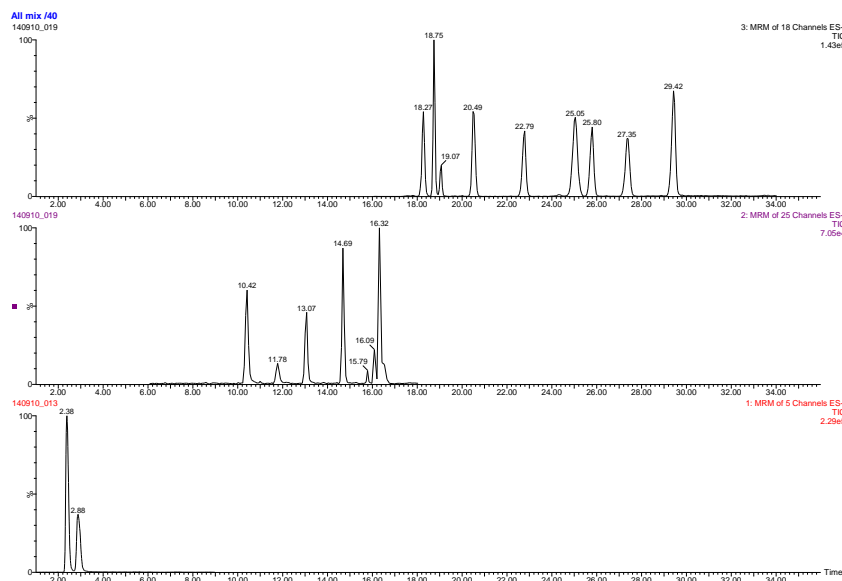


Figure 3: LC-MS² chromatogram of a standard mixture (negative ionization).

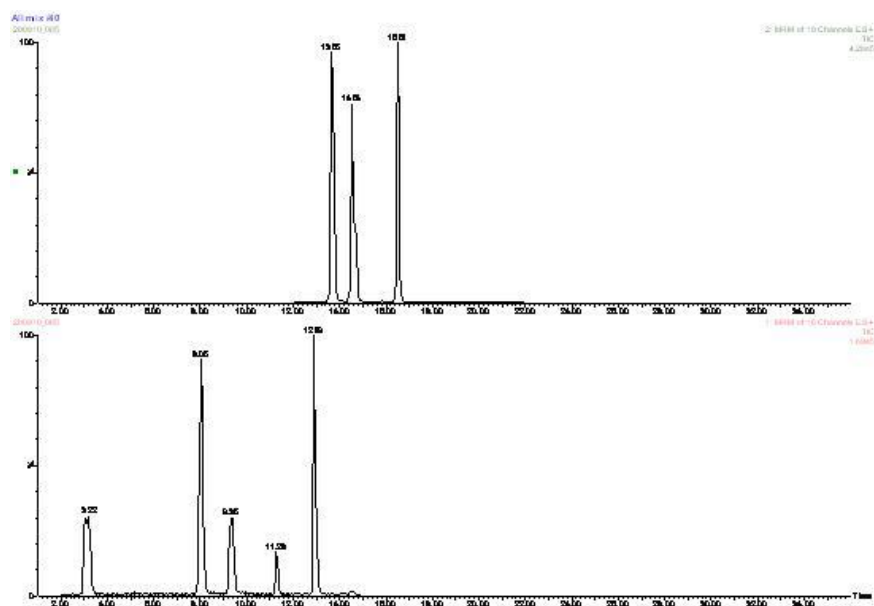


Figure 4: LC-MS² chromatogram of a standard mixture (positive ionization).

The analytical parameters MRM transitions, retention times, recoveries, LOQs, and the internal standards used for the quantification are given in Table 10.

In comparison to the analysis of surface and ground water (Loos et al., 2009a, 2010), the LOQs for the analysis of waste water were a bit higher, due to higher matrix interferences and lower recoveries.

6.2. Chemical compounds identified

Table 19 summarizes the analytical results for the polar organic compounds which were measured in the WWTP effluents across Europe and all single analytical results are given in Annex 2-10. In total 90 effluent samples were analysed (by JRC-IES). The UMEÅ University (Sweden) analysed 89 samples, UBA (Austria) 77 samples, IWW (Germany) and Masaryk University 73 samples, and VITO (Belgium) 30 samples. The compounds are sorted according to their frequency of detection (Freq. in %); “Max.” is the maximum concentration found, “Med.” the median concentration, and “Per90” the 90th percentile.

Table 19: Summary of analytical results for chemicals in EU WWTP effluents (Fate Sees).

Chemical	LOQ [ng/l]	Freq. [%]	Max. [ng/l]	Average [ng/l]	Med. [ng/l]	Per90 [ng/l]
TBEP, Tris(2-butoxyethyl)phosphate	1	100	43 µg/l	2220,3	190,0	7580,0
TCPP, Tris(2-chloroisopropyl)-phosphate	1	100	21 µg/l	1231,2	620,0	2100,0
Irbesartan	0,5	100	17.9 µg/l	479,5	85,4	859,9
DEET, N,N'-Diethyltoluamide	1	100	15.8 µg/l	678,1	195,8	1091,3
TCEP Tris(2-chloroethyl)phosphate	1	100	2400,0	130,9	71,0	246,0
TBP, Tributylphosphate	1	100	1700,0	259,8	160,0	574,0
Tramadol	0,5	100	1165,7	255,8	218,4	503,5
TIBP, Tri-iso-butylphosphate	1	100	870,0	133,2	110,0	244,0
TDCP, Tris(1,3-dichloro-2-propyl)phosphate	1	100	860,0	176,2	110,0	480,0
Gadolinium	1,9	100	789,4	115,0	58,4	236,4
TPP, Triphenylphosphate	1	100	610,0	35,6	17,0	71,8
Risperidone	0,1	100	85,8	6,9	3,3	15,6
Trihexyphenidyl	0,1	100	1,7	0,2	0,0	0,4
Methylbenzotriazole	40	100	24,3 µg/l	2,9 µg/l	2,1 µg/l	4,7 µg/l
PFOA, Perfluorooctanoic acid	1	99	15.9 µg/l	255,0	12,9	60,9
Venlafaxin	0,5	99	548,3	118,9	97,0	242,6
Codeine	0,5	98	826,4	70,6	20,9	167,0
Fluconazole	0,5	98	598,1	108,2	67,5	286,7
Diphenhydramin	0,05	98	141,5	11,7	4,9	26,0
Repaglinide	0,5	98	12,3	3,1	2,1	6,6
1H-Benzotriazole	40	97	221 µg/l	6,3 µg/l	2,7 µg/l	10,9 µg/l
Flecainide	0,1	97	552,6	45,5	10,8	108,6
Bisoprolol	0,1	97	423,4	41,6	15,7	135,6
EHDPP, 2-Ethylhexyldiphenyl-phosphate	1	94	5400,0	92,7	6,5	42,2
PFHpA, Perfluoroheptanoic acid	1	94	2962,3	82,9	5,1	20,3

PFOS, Perfluorooctansulfonate	0,5	93	2100,9	62,5	12,2	94,1
Trimethoprim	10	93	799,8	229,0	178,3	552,4
Acesulfame K	10	93	2.5 mg/l	76 µg/l	14,3 µg/l	61 µg/l
Caffeine	5	93	3002,0	191,1	34,6	400,0
Alfuzosin	0,1	91	20,5	2,8	1,5	8,3
Bupropion	0,1	91	4,9	1,0	0,6	2,7
Ciprofloxacin	15	90	264,5	96,3	82,1	197,1
Oxazepam	5	90	1765,8	161,7	64,3	429,0
Carbamazepine	1	90	4608,8	832,3	751,9	1556,4
Diclofenac ^{*1}	1	89	174,3	49,5	43,3	102,8
PFNA, Perfluorononanoic acid	0,5	89	2734,5	35,1	2,3	7,9
Sucralose	100	88	12,9 µg/l	2,6 µg/l	1,7 µg/l	6,1 µg/l
Orphenadrin	0,1	85	46,7	3,9	0,5	6,8
Sulfamethoxazole (VITO)	20	83	1690,5	280,2	164,1	617,7
Haloperidol	0,1	83	2691,5	32,2	0,3	1,2
Citaprolam	5	83	188,6	33,8	21,1	74,6
PFDA, Perfluorodecanoic acid	1	81	1687,0	23,9	2,9	7,7
Sulfamethoxazole (JRC)	1	81	1147,4	142,3	67,5	398,7
Fexofenadine	5	80	1287,1	165,0	58,8	431,3
MCPA	0,5	80	2403,9	149,9	22,1	499,0
Diltiazem	0,5	79	64,4	10,7	6,4	25,7
Fluoxetine	5	78	21,5	2,1	0,0	7,8
Diuron	1	77	1425,8	61,7	11,6	83,7
Terbutalin	0,5	76	4,7	1,1	0,9	2,6
Clindamycine	10	73	277,2	70,4	45,9	150,9
PFHxA	1	72	23866,5	303,7	5,7	18,2
2,4-D	1	72	356,7	27,1	11,9	72,2
Mecoprop	1	72	2208,6	127,4	17,2	251,7
Diazinon	1	71	391,0	21,4	4,1	30,1
PFHxS, Perfluorohexansulfonate	1	71	921,9	48,6	3,4	34,8
Terbutylazine	1	67	2411,4	90,6	4,7	96,9
Atrazine	1	68	36,6	4,2	2,2	11,0
Naproxen	5	66	957,6	26,7	8,2	33,1
Telmisartan	50	63	4344,8	367,5	120,1	888,6
Bezafibrate	1	63	343,5	25,4	3,5	81,9
Eprosartan	5	62	6810,2	226,8	13,7	432,5
Gemfibrozil	1	60	3618,5	137,7	4,9	300,4
Zolpidem	0,5	58	42,6	1,5	0,6	1,7
Ibuprofen	2	57	2128,5	80,5	7,0	134,2
Hydroxyzine	0,5	51	9,6	1,1	0,5	3,0
Terbutylazine-desethyl	3	51	1530,8	68,8	4,7	131,1
Isoproturon	1	49	270,4	10,1	0,4	19,5
Ketoprofen	10	49	1653,1	86,0	0,0	179,7
Bentazone	1	48	220,0	9,6	0,0	20,6
Amidotrizoic acid	50	47	8,4 µg/l	619	0,0	1,7 µg/l
Ranitidine	5	42	43,6	6,8	0,0	21,5
Nitrophenol	5	42	1300,8	49,4	0,0	84,6
Triclosan ^{*2}	4	41	4258,6	74,8	0,0	100,5
Levamisol	15	37	339,9	40,6	0,0	116,0
Lincomycine	15	37	317,1	31,2	0,0	106,5
Rosuvastatin	10	36	978,9	31,0	0,0	41,6

Iopromid	50	36	150 µg/l	2,7 µg/l	0,0	1,3 µg/l
Biperiden	0,1	35	2,4	0,1	0,0	0,3
Penicilline V	50	33	122,0	28,7	0,0	97,3
Clotrimazol	1	33	5,3	0,6	0,0	2,0
Metolachlor	3	29	288,9	12,4	0,0	38,0
Dichlorprop	3	29	186,3	9,6	0,0	19,2
Mianserin	1	28	62,3	1,5	0,0	3,8
Simazine	5	28	689,3	26,3	0,0	67,1
Clofibrac Acid	2	26	127,0	5,3	0,0	12,1
Dinitrophenol	5	26	473,5	18,8	0,0	58,0
Atrazine-desethyl	3	24	155,4	13,8	0,0	54,6
Iomeprol	50	22	12,0 µg/l	376	0,0	378
Clomipramine	0,5	20	3,0	0,3	0,0	1,0
Iohexol	50	18	7,7 µg/l	158	0,0	258
Iopamidol	50	15	6,1 µg/l	144	0,0	158
Memantin	0,5	12	1312,1	22,8	3,7	23,1
Sertraline	10	12	37,5	2,1	0,0	11,1
Tiamuline	20	10	44,5	3,3	0,0	2,4
Clonazepam	5	9	43,7	1,6	0,0	0,0
Chlortoluron	3	9	183,7	3,2	0,0	0,0
Carbaryl	3	9	81,4	1,6	0,0	0,0
Hexazinon	2	9	42,0	0,8	0,0	0,0
Loperamide	0,5	8	2409,1	29,3	0,0	0,0
Alprazolam	10	8	33,0	1,3	0,0	0,0
Pizotifen	0,5	8	1,9	0,1	0,0	0,0
Fenofibrate	10	7	25,7	1,1	0,0	0,0
Nefazodon	0,5	7	2,4	0,1	0,0	0,0
Linuron	5	7	3264,8	40,1	0,0	0,0
Buprenorphin	10	6	259,1	3,9	0,0	0,0
Fentanyl	0,5	6	1,6	0,1	0,0	0,0
Methabenzthiazuron	3	6	17,8	0,5	0,0	0,0
Cilazapril	1	4	2,2	0,1	0,0	0,0
Maprotilin	5	3	16,5	0,4	0,0	0,0
Sulfadiazine	30	3	105,4	3,5	0,0	0,0
Tilmicosine	50	3	93,2	3,1	0,0	0,0
Flumequine	20	3	25,7	0,9	0,0	0,0
2,4,5-T	2	3	10,2	0,3	0,0	0,0
Cyproheptadine	5	2	325,1	3,9	0,0	0,0
Glibenclamide	10	2	28,0	0,4	0,0	0,0
Amitryptiline	5	2	14,6	0,3	0,0	0,0
Tamoxifen	5	2	12,6	0,3	0,0	0,0
Duloxetine	1	2	6,3	0,1	0,0	0,0
TCP, Tricresylphosphate	1	2	1,3	0,0	0,0	0,0
Atorvastatin	59	1	72,9	0,8	0,0	0,0
Verapamil	10	1	16,5	0,2	0,0	0,0
Miconazole	5	1	14,8	0,2	0,0	0,0
Chlorpromazine	5	1	10,4	0,1	0,0	0,0
Flupetixol	5	1	7,9	0,1	0,0	0,0
Flutamid	5	1	5,8	0,1	0,0	0,0

Substances not found: Azelastine, Bromocriptin, Chloprothixen, Clemastine, Dicycloverin, Etonogestrel, Fluphenazine, Glimepiride, Meclozine, Paroxetin, Perphenazine, Promethazin, TEHP, Oxytetracycline, Doxycycline, Penicilline G, Amoxilline, Ampicilline, Florfenicol, Tylosine, Sulfadoxine, Enrofloxacin, Fenbendazole, Decamethyltetrasiloxan (MD2M), Dodecamethylpentasiloxan (MD3M), Octamethyltrisiloxan (MDM), Celestolid, Phantolid, and Traesolid.

*¹Diclofenac: It might be possible that the levels for Diclofenac are higher than reported here (see section 6.3.3.)

*²Triclosan levels are probably the double of the reported concentrations because the used HDPE bottles are not recommended for use when sampling for this compound (due to adsorption) (Vanderford et al., 2012, chapter 2, page 19).

6.2.1. Frequency of detection

The high overall frequency of detection (above the LOQ) is shown by the percentile frequency (freq.) of detection for the compounds. The average frequency of detection for all 161 compounds was 43%.

The most frequently detected compounds were DEET, Methylbenzotriazole, Irbesartan, Risperidone, Tramadol, Trihexyphenidyl, TIBP, TBP, TCEP, TCPP, TDCP, TBEP, TPP, Gadolinium (all 100% frequency), PFOA, Venlafaxin (99%), Codeine, Diphenhydramin, Fluconazole, Repaglinide (98%), Benzotriazole, Bisoprolol, Flecainide (97%), EHDPP, PFHpA (94%), PFOS, Trimethoprim, Acesulfame K, Caffeine (93%), Alfuzosin, Bupropion (91%), Ciprofloxacin, Oxazepam, Carbamazepine (90%), Diclofenac, PFNA (89%), Sucralose (88%), Orphenadrin (85%), Citaprolam, Haloperidol (83%), PFDA, Sulfamethoxazole (81%), Fexofenadine, MCPA (80%), Diltiazem (79%), etc. (Table 19 and Figure 5).

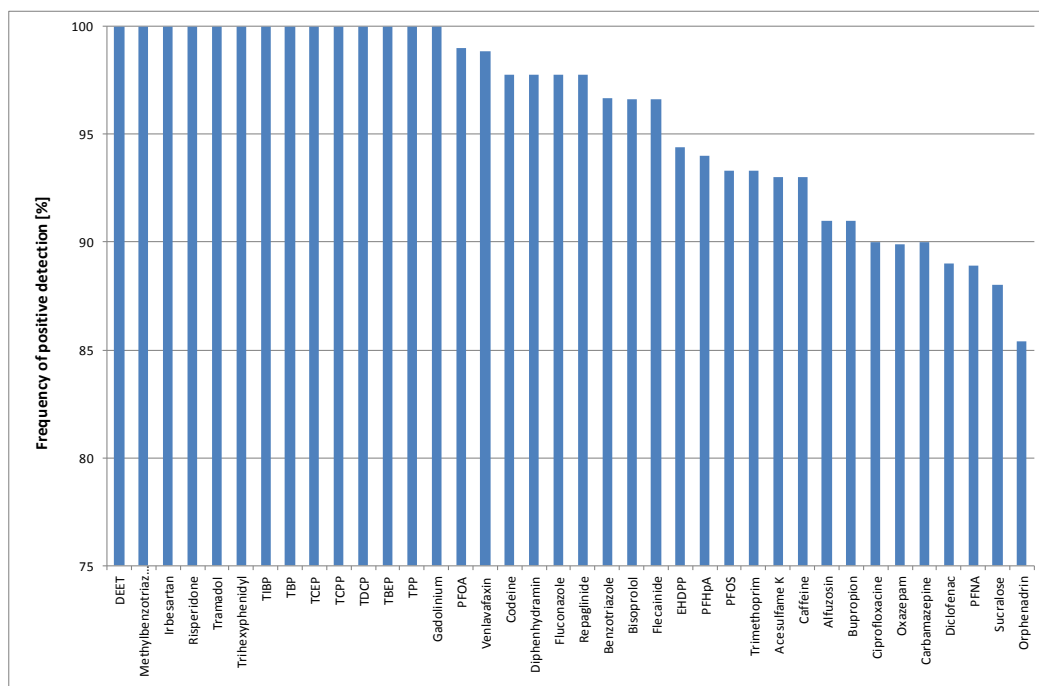


Figure 5: Most frequently detected compounds.

Twenty eight compounds (out of 161) were not detected in any of the samples. These substances were: Azelastine, Bromocriptin, Chloprothixen, Clemastine, Dicycloverin, Etonogestrel, Fluphenazine, Glimepiride, Meclozine, Paroxetine, Perphenazine, Promethazine, TEHP, Oxytetracycline, Doxycycline, Penicilline G, Amoxicillin, Ampicillin, Florfenicol, Tylosine, Sulfadoxine, Enrofloxacin, Fenbendazole, Decamethyltetrasiloxan (MD2M), Dodecamethylpentasiloxan (MD3M), Octamethyltrisiloxan (MDM), Celestolid, Phantolid, and Traesolid.

It must be noted that also 17β -Estradiol, 17α -Ethinylestradiol, and Estrone were analysed within the multi-compound screening method (negative ESI method) in all samples (by [JRC-IES](#)), but these estradiol hormones were not detected in any of the samples above the LOQ of ~ 10 ng/l.

Other substances with a low frequency of detection ($<10\%$) were Verapamil, Miconazole, Flutamid, Flupetixol, Chlorpromazine, Atorvastatin, Tonalid, Octamethylcyclotetrasiloxan (D4), Dodecamethylcyclohexasiloxan (D6), Decamethylcyclopentasiloxan (D5), 2,4,5-T, TCP, Tamoxifen, Glibenclamide, Duloxetine, Cyproheptadine, Amitriptyline, Flumequine, Sulfadiazine, Tilmicosine, Maprotilin, Cilazapril, Methabenzthiazuron, Fentanyl, Buprenorphin, Linuron, Nefazodon, Fenofibrate, Pizotifen, Loperamide, Alprazolam, Chlortoluron, Hexazinon, Carbaryl, and Clonazepam (see Table 19).

These chemicals have a relatively low relevance for occurrence in the effluents of WWTPs.

6.2.2. Maximum concentrations

The highest maximum concentrations were found for the chemicals Acesulfame K (2.5 mg/l), 1H-Benzotriazole (221 μ g/l), Iopromid (150 μ g/l), TBEP 43 μ g/l, Methylbenzotriazole (24 μ g/l), PFHxA (23.9 μ g/l), TCPP (21 μ g/l), Irbesartan (17.9 μ g/l), PFOA (15.9 μ g/l), DEET (15.8 μ g/l), Sucralose (12.9 μ g/l), Iomeprol (12.0 μ g/l), Amidotrizoic acid (8.4 μ g/l), Iohexol (7.7 μ g/l), Eprosartan (6.8 μ g/l), Iopamidol (6.1 μ g/l), EHDPP (5.4 μ g/l), Carbamazepine (4.6 μ g/l), Telmisartan (4.3 μ g/l), Triclosan (4.3 μ g/l), Gemfibrozil (3.6 μ g/l), Linuron (3.2 μ g/l), Caffeine (3.0 μ g/l), PFHpA (3.0 μ g/l), PFNA (2.7 μ g/l), Haloperidol (2.7 μ g/l), Terbutylazine (2.4 μ g/l), Loperamide (2.4 μ g/l), MCPA (2.4 μ g/l), TCEP (2.4 μ g/l), Mecoprop (2.2 μ g/l), Ibuprofen (2.1 μ g/l), PFOS (2.1 μ g/l), Oxazepam (1.8 μ g/l), TBP (1.7 μ g/l), Sulfamethoxazole (1.7 μ g/l), PFDA (1.7 μ g/l), Ketoprofen (1.7 μ g/l), Terbutylazine-desethyl (1.5 μ g/l), Diuron (1.4 μ g/l), Memantin (1.3 μ g/l), etc. (see Table 19 and Figure 6).

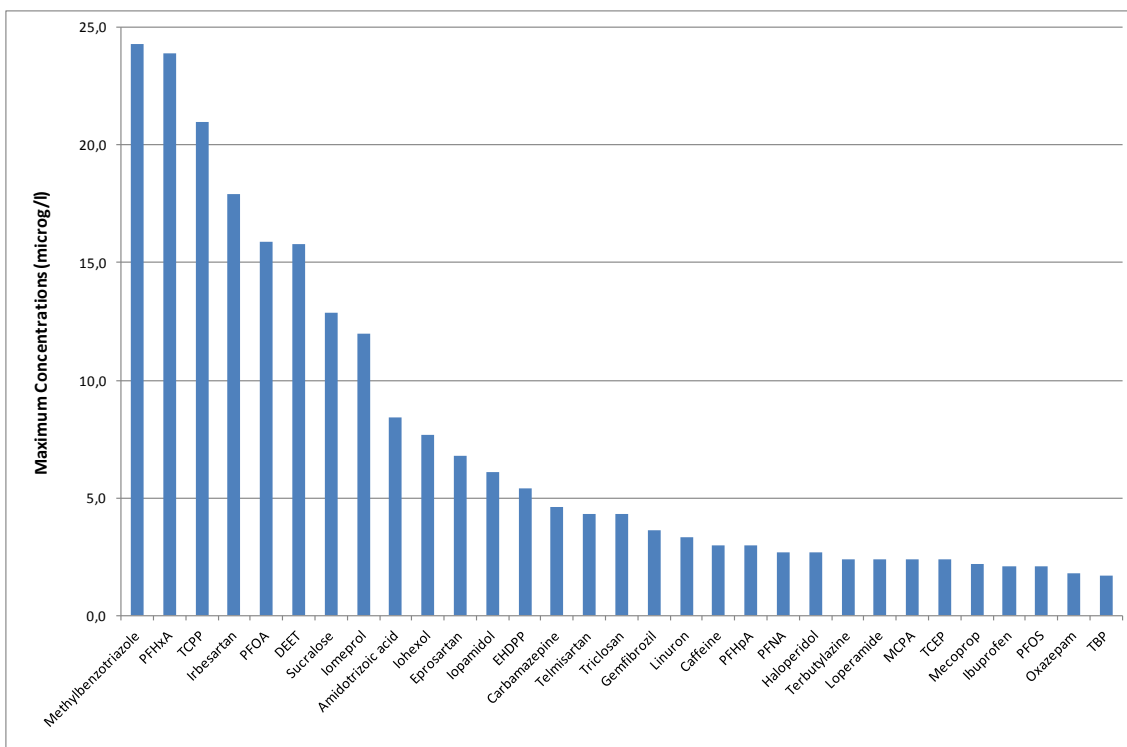


Figure 6: Chemicals found at the highest concentrations. Excluded are Acesulfame K (2.5 mg/l), 1H-Benzotriazole (221 µg/l), lopromid (150 µg/l), and TBEP (43 µg/l).

6.2.3. Median concentrations

Table 19 shows also the median (med.) concentration levels for the substances studied. The highest median concentrations were measured for Acesulfame K (14.3 µg/l), 1H-Benzotriazole (2.7 µg/l), Tolyltriazole (2.1 µg/l), Sucralose (1.7 µg/l), Carbamazepine (752 ng/l), TCPP (620 ng/l), Tramadol (218 ng/l), DEET (196 ng/l), TBEP (190 ng/l), Trimethoprim (178 ng/l), TBP (160 ng/l), Telmisartan (120 ng/l), TIBP (110 ng/l), TDCP (110 ng/l), Venlafaxin (97 ng/l), Irbesartan (85 ng/l), Ciprofloxacin (82 ng/l), TCEP (71 ng/l), Fluconazole (68 ng/l), Sulfamethoxazole (68 ng/l), Oxazepam (64 ng/l), Fexofenadine (59 ng/l), Gadolinium (58 ng/l), Clindamycin (46 ng/l), Caffeine (35 ng/l), MCPA (22 ng/l), Citaprolam (21 ng/l), and Codeine (21 ng/l). These results show the high relevance of these substances in effluents (Figure 7).

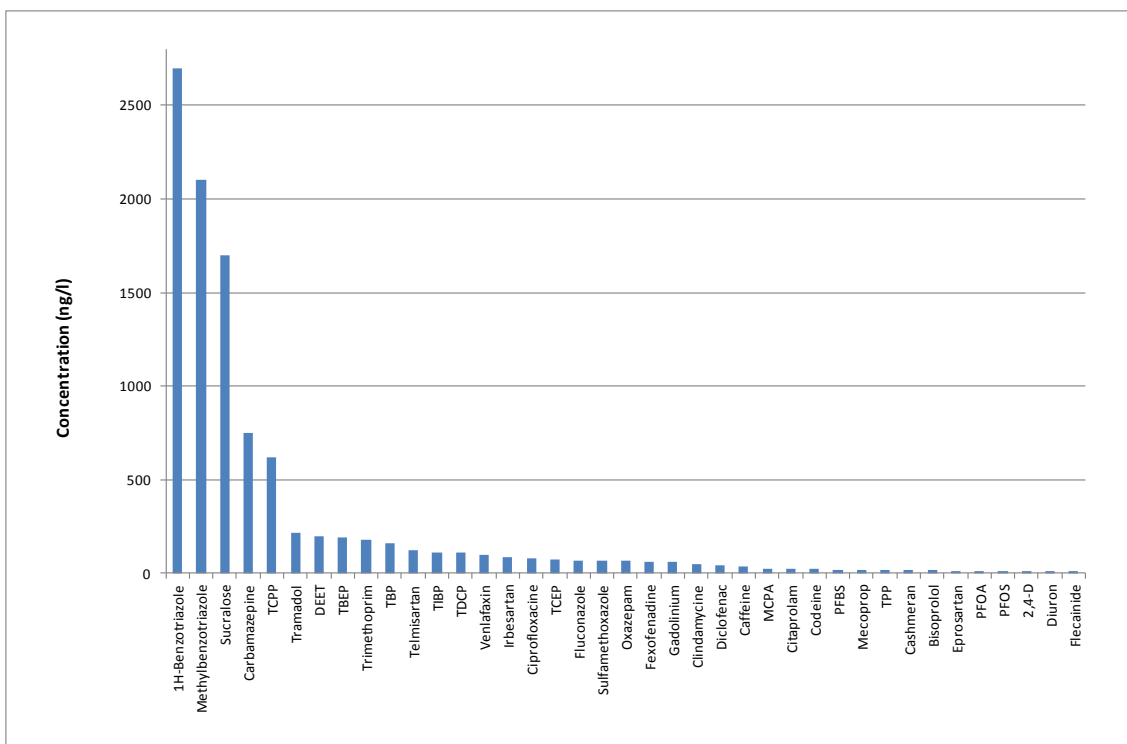


Figure 7: Median concentrations of the target compounds (90 samples).

6.3. Comparison with other WWTP studies (Interpretation)

In the EU-wide WWTP study performed by [Reemtsma and co-workers \(2006\)](#) (see introduction), EDTA (Ethylenediaminetetraacetic acid), a complexing agent, (which was not analysed in Fate Sees) was the substance with by far the highest median concentrations (60 µg/l). Moreover, very high concentrations (median: 57 µg/l) were found in this study for sulfophenyl carboxylates, a group of biodegradation intermediates of the anionic surfactants LAS (linear alkylbenzene sulfonates). In our study (Fate Sees), the highest median concentration was found for Acesulfame K (14.3 µg/l), followed by the Benzotriazoles. The median concentration found for 1H-Benzotriazole (2.7 µg/l) corresponds very well to the study by [Reemtsma et al. \(2006\)](#), who found a median of 2.9 µg/l.

Other compounds determined by [Reemtsma et al. \(2006\)](#) at relatively high levels (µg/l range) were Benzothiazoles, Naphthalenedisulfonates, and Nonylphenolethoxycarboxylates (NPECs), the predominant biodegradation intermediates of NPEO (Nonylphenolethoxylates) surfactants (not analysed in Fate Sees). For many pharmaceuticals (Diclofenac, Ibuprofen, Ketoprofen, Naproxen, Bezafibrate, Carbamazepine, Clofibric acid, and Sulfmethoxazole) median concentrations in the range of 0.1-1 µg/l in WWTP effluents were found in the study by [Reemtsma and co-workers \(2006\)](#).

In the Swiss “Micropoll Strategy” project (Micropoll, 2011; 2012), 47 Swiss-specific micropollutants were identified out of 250 candidate substances (using a prioritization procedure), representative for the contamination caused through micropollutants from municipal wastewater. Table 20 shows the average effluent concentrations for these substances in comparison to the Fate Sees results; they are discussed in the following parts.

Table 20: Comparison of the Fate Sees data with data from the Swiss Micropoll project; average effluent concentrations in ng/l; n = number of measurements; n.a. = not analysed.

	Micropoll Project (CH)			Fate Sees (Europe)		
	Average	n pos.	n	Average	n	Freq.(%)
Amidotrizoic acid (contrast agent)	598	7	10	619	73	57
Iomeprol (contrast agent)	380	9	19	376	73	37
Iopamidol (contrast agent)	377	15	19	144	73	31
Iopromid (contrast agent)	876	13	19	2680	73	48
Acesulfame K	22500	4	4	76100	90	93
Sucralose	4600	6	6	2600	90	87
Benzotriazole	12881	41	41	6300	90	97
Methyl-benzotriazole	1140	30	30	2900	90	99
Bezafibrate	139	12	15	25	90	62
Carbamazepine	482	78	78	832	90	89
Diclofenac	647	54	54	50	90	90
Ibuprofen	394	54	54	81	90	56
Naproxen	462	38	39	27	90	64
Sulfamethoxazole	238	34	34	142	90	81
Trimethoprim	100	42	45	229	30	93
Triclosan	116	6	6	75	90	41
PFOS	114	7	7	63	90	93
2,4-D	13	4	6	27	90	71
Diazinon (insecticide)	173	40	84	21	90	71
DEET (insect repellent)	593	11	55	678	90	100
Diuron	1379	13	34	62	90	77
Isoproturon (herbicide)	12	11	14	10	90	53
MCPA	25	6	6	150	90	79
Mecoprop	424	26	29	127	90	69
Carbamazepine-10,11-dihydro-10,11-dihydroxy	1551	6	6	n.a.		
Atenolol (betablocker)	843	18	18	n.a.		
Metoprolol (betablocker)	166	17	17	n.a.		
Sotalol (betablocker)	435	21	21	n.a.		
Azithromycin (antibiotic)	175	18	19	n.a.		
Clarithromycin (antibiotic)	276	32	32	n.a.		
Erythromycin (antibiotic)	42	17	17	n.a.		
Mefenamic acid (analgetic)	870	10	10	n.a.		
Metformin (antidiabetikum)	10347	6	6	n.a.		
Irgarol (herbicide)	30	9	29	n.a.		
Carbendazim (fungicide)	81	17	30	n.a.		
Bisphenol A	331	22	25	n.a.		
Estradiol (hormone)	3	18	28	< 5		
Ethinylestradiol (hormone)	2	6	27	< 5		

Nonylphenol	267	7	7	n.a.		
Benzothiazol (additive)	494	6	6	n.a.		
EDTA (complexing agent)	20930	10	10	n.a.		
NTA (complexing agent)	5370	10	10	n.a.		

6.3.1. Perfluoroalkyl Substances (PFASs)

Polyfluorinated compounds (PFCs) are useful anthropogenic chemicals that have been incorporated into a wide range of products for the past six decades. The products in which they are utilized include protective coatings for food contact packaging, textiles, carpets, paper, coats, fabrics, leather, non-stick cooking material, commercial and industrial surfactants (e.g. fire-fighting foams, electroplating baths), and insecticides. This class of compounds includes thousands of chemicals, but is best known for the perfluorosulfonates (PFSAs) such as perfluorooctane sulfonate (PFOS), and the perfluorocarboxylic acids (PFCAs) which include perfluorooctanoic acid (PFOA). Concerns about these compounds have developed as many satisfy the defining characteristics of persistent organic pollutants (POPs): they are toxic, extremely resistant to degradation, bioaccumulate in food chains, are transported over large distances, and can have long half-lives in humans. They are ubiquitous present in the environment and wildlife, and further have been found in human blood serum worldwide ([Lindstrom et al., 2011](#)).

The discharge of municipal waste waters is one of the principal routes of entry of PFOA and PFOS into the aquatic environment ([Becker et al., 2008](#); [Guo et al., 2010](#); [Huset et al., 2008](#); [Loganathan et al., 2007](#); [Ma and Shih, 2010](#); [Schultz et al., 2006a,b](#); [Sinclair and Kannan, 2006](#)). Often PFAS concentrations increase in WWTPs as a result of biodegradation of precursors during the activated sludge process. In Bayreuth, Germany, it was found that PFOA is fully discharged into the river, while about half of PFOS is retained in the sewage sludge ([Becker et al., 2010](#)). Despite the voluntary phasing out of the production of perfluorooctane sulfonyl-based chemicals in 2002 (by the main producers), the detection of PFOS in WWTPs indicates that products containing PFASs are still releasing these substances into the environment.

The most frequent PFAS found in the Fate Sees study was PFOA (detection frequency of 99%), followed by PFOS (93%), PFHpA (C7; 94%), PFNA (C9; 89%), PFDA (C10; 81%), PFHxA (C6; 71%), and PFHxS (C6; 70%). PFOA was also detected at the highest median concentration levels (12.9 ng/l), followed by PFOS (12.2 ng/l), PFHxA (5.7 ng/l), PFHpA (5.1 ng/l), PFHxS (3.4 ng/l), PFDA (2.9 ng/l), and PFNA (2.3 ng/l). The highest (single) maximum concentrations were found for PFHxA (23.9 µg/l), and PFOA (15.9 µg/l), followed by PFHpA (3.0 µg/l), PFNA (2.7 µg/l), PFOS (2.1 µg/l), PFDA (1.7 µg/l), and PFHxS (922 ng/l).

Similar PFAS levels are reported in the literature (Table 21). Highest maximum levels were found in the Fate-Sees study in industrial WWTPs. In WWTPs in Bayreuth (Germany), PFOA was found in all treated waste water samples in concentrations ranging from 9 to 93 ng/l, and PFOS at levels from 12–336 ng/l ([Becker et al. 2008; 2010](#)). In

Denmark, levels in 11 WWTPs were for PFOA <2-115.4 ng/l (median: 16.9 ng/l), and for PFOS <1-1115 ng/l (median: 4.3 ng/l) (Bossi et al., 2008). In six WWTPs in New York (USA) PFOS effluent concentrations ranged between 3-68 ng/l, and between 58-1050 ng/l for PFOA (Sinclair and Kannan, 2006). Guo and co-workers (Guo et al., 2010) investigated the fate of PFASs in WWTPs in Korea. Ten PFASs were analyzed in influent and effluent wastewater and sludge samples in 15 municipal, 4 livestock and 3 industrial WWTPs. The observed distribution pattern of PFASs differed between the wastewater and sludge samples. PFOS was dominant in the sludge samples with a concentration ranging from 3.3-54 ng/g, whereas PFOA was dominant in wastewater and ranged from 2.3 to 615 ng/l and 3.4 to 591 ng/l in influent and effluent wastewater, respectively. PFOS was the second dominant compound in the wastewater with the level of not detected (n.d.)-68 ng/l and n.d.-8.9 ng/l in the influent and effluent, respectively. Concentrations of PFOS in the effluent were 4- to 70-fold lower than those of WWTPs (42-635 ng/l) in Japan (Murakami et al., 2009), but similar to those of WWTPs in Kentucky and Georgia, USA (1.8-28 ng/l) (Loganathan et al., 2007). Very high PFOA effluent concentrations were reported from China in industrial WWTPs (Chen et al., 2012a).

Table 21: Overview of PFOA and PFOS concentrations (ng/l) in WWTP effluents.

	Number of WWTPs	PFOA	PFOS	Reference
Fate Sees	90	<1-15940 Median: 12.9	<0.5-2101 Median: 12.2	This study
USA	1	8.2-15	15-34	Schultz et al. (2006b)
USA (New York)	6	58-1050	3-68	Sinclair and Kannan (2006)
USA (Kentucky, Georgia)	2	6.7-183	1.8-28	Loganathan et al. (2007)
Denmark	11	<2-115.4 Median: 16.9	<1-1115 Median: 4.3	Bossi et al. (2008)
Switzerland	4	12-35	16-303	Huset et al. (2008)
Germany	9	12-78	0.06-82	Ahrens et al. (2009)
Japan	5	10-68	42-635	Murakami et al. (2009)
Singapore	13	16-1057	7.3-462	Yu et al. (2009b)
Germany (Bayreuth)	4	9-93	12-336	Becker et al. (2008, 2010)
Korea	22	3.4-591	n.d.-8.9	Guo et al. (2010)
Hong Kong	4	n.d.-4.1	19-50	Ma and Shih (2010)
China (Taiwan)	2	19-25	163-265	Lin et al. (2010)
China	12	Domestic: 3-191 Ind.: 160-280 µg/l	1-75	Chen et al. (2012a)

6.3.2. Benzotriazoles

1H-Benzotriazole (BT) and Methyl-Benzotriazoles (=Tolyltriazole, TT, used as a technical mixture of 4- and 5-TT) are a class of high production volume chemicals which find broad applications in various industrial processes as well as in households. They show metal complexing properties and are used as anticorrosive additives and flame retardants in aircraft de-icers and anti-ice fluids, in cooling and hydraulic fluids and for silver protection in dishwashing agents (Janna et al., 2011; Reemtsma et al., 2010; Weiss

et al., 2006). Benzotriazoles are compounds with high water solubility and a high polarity. Moreover, they are quite persistent against biological and photochemical degradation processes in the aquatic environment (Hart et al., 2004). Acute toxicity to aquatic organisms is in the low to moderate mg/l range, but there is a lack of a complete set of good quality eco-toxicological data on possible chronic effects of these high use chemicals (Janna et al., 2011).

Benzotriazoles were for the first time analysed in Berlin (Germany) and Switzerland. In Switzerland, BT and TT were found in the $\mu\text{g/l}$ range in all samples of primary and secondary effluents from 24 municipal WWTPs. The BT concentrations were always by a factor of 10 to 100 higher than the corresponding TT values. The median values for BT concentrations in primary and secondary effluents were 18 and 10 $\mu\text{g/l}$, respectively (Voutsas et al., 2006). Four WWTPs in Berlin, treating municipal wastewater as a mix of household and industrial wastewater were investigated for the occurrence and removal of benzotriazoles over a six month period. Mean influent concentrations for the four plants ranged from 17-44 $\mu\text{g/l}$ for BT and from 1.1-4.9 $\mu\text{g/l}$ for each of the TT-isomers. Of the three benzotriazoles, none was eliminated completely during wastewater treatment. Generally, relative removal was best for 5-TT, but highly variable between the plants; it ranged from 19 to 69%. BT showed moderate removal (29–58%), whereas for 4-TT only one plant showed a significant but still limited removal (34%). Owing to the incomplete removal, BT median values of 7–18 $\mu\text{g/l}$ were found for the effluents of the four WWTP (Reemtsma et al., 2010). In a previous study on WWTPs in Berlin 12 $\mu\text{g/l}$ for BT and 1.3 $\mu\text{g/l}$ for 5-TT and 2.1 $\mu\text{g/l}$ for 4-TT were reported as the two-year average (Weiss et al., 2006). Concentrations of 1H-Benzotriazole in eight UK sewage effluents ranged from 840 to 3605 ng/l, and were consistently less than those of TT (2685 to 5700 ng/l; in contrary to Germany and Switzerland). There was no relationship between effluent concentrations and treatment type, which were either activated sludge or biological filters with or without tertiary treatment (Janna et al., 2011). It is clear that the occurrence of Benzotriazoles in municipal wastewaters is caused by its application as an anticorrosive additive in dishwasher products, but there are also many industrial applications.

In our Fate-Sees study median concentrations were 2.7 $\mu\text{g/l}$ for BT, and 2.1 $\mu\text{g/l}$ for 5-TT, with maximum values found up to 221 $\mu\text{g/l}$ and 24.3 $\mu\text{g/l}$ for BT and 5-TT, respectively. These levels are in relatively good agreement with the other studies cited above. Similarly to the studies from Berlin and Switzerland, higher levels were found for BT.

Table 22: Comparison of the Fate Sees data for Benzotriazoles with other studies; WWTP effluent concentrations in (µg/l).

Location	Reference	(µg/l)
Fate Sees (Europe; n=90)	This report	1H-Benzotriazole: Max.: 221 Average: 6.3 Median: 2.7 Methyl-Benzotriazole: Max.: 24.3 Average: 2.9 Median: 2.1
10 WWTPs (CH)	Voutsas et al. (2006)	1H-Benzotriazole: Median: 10
WWTPs (Berlin, Germany) (two years average)	Weiss et al. (2006)	1H-Benzotriazole: Median: 12 4-Methyl-Benzotriazole: Median: 2.1 5-Methyl-Benzotriazole: Median: 1.3
WWTPs in western Europe	Reemtsma et al. (2006)	1H-Benzotriazole: Median: 6.6 Methyl-Benzotriazole: Max.: 0.7
4 WWTPs (Berlin, Germany)	Reemtsma et al. (2010)	1H-Benzotriazole: Median: 7-18
WWTP Vidy Lausanne (CH)	De la Cruz et al. (2012)	1H-Benzotriazole: Average: 2.8 Methyl-Benzotriazole: Average: 1.5

6.3.3. Pharmaceuticals

The removal of pharmaceuticals (or PPCPs) and other micropollutants in WWTPs has been investigated in several excellent studies before. State of the art biological treatment schemes for municipal wastewater are not efficient in degrading many pharmaceutical compounds; they are only poorly removed in conventional WWTPs.

For example, Joss and coworkers (2005) studied the removal of seven pharmaceuticals and two fragrances in the biological units of various full-scale municipal WWTPs. The observed removal of pharmaceuticals was mainly due to biological transformation and varied from insignificant (<10% for Carbamazepine and Sulfamethoxazole) to >90% (Ibuprofen). Ibuprofen is considered to be readily biodegradable, and removal rates reported in the literature are in the range of 80-100%. Stronger fluctuations were observed for other substances (e.g. Bezafibrate) (Clara et al., 2005; Lindqvist et al., 2005; Hollender et al., 2009; Quintana et al., 2005; Radjenović et al., 2007a). Naproxen also showed significant removal (50–80%), and partial or contradictory removal was seen for Diclofenac (10–70%) (Joss et al, 2005; Radjenović et al., 2007a). In a subsequent study, Joss and coworkers studied a heterogeneous group of

35 compounds, and found that only 4 out of 35 compounds were degraded by more than 90% while 17 compounds were removed by less than 50% (Joss et al, 2006).

Carbamazepine and Diclofenac are the most frequently detected pharmaceutical residues in water bodies thus far, since they are poorly removed in WWTPs (Zhang et al., 2008). Investigations found that Carbamazepine is persistent and its removal efficiencies by the WWTPs are mostly below 10%, because it is resistant to biodegradation at low concentrations, and it is hardly attached onto sludge. The removal efficiencies of Diclofenac by WWTPs vary, ranging from 0 up to 80%, but mainly in the scope of 20–40%. It has a relatively low biodegradation potential, but biodegradation is possible under some conditions. In any case, Diclofenac is less persistent than Carbamazepine, and the sorption behavior onto sludge is similar to that of Carbamazepine. Phototransformation of Diclofenac, however, is much faster than for Carbamazepine (Zhang et al., 2008).

Lindqvist and coworkers (2005) studied the five acidic pharmaceuticals Ibuprofen, Naproxen, Ketoprofen, Diclofenac and Bezafibrate in seven different WWTPs in Finland. In the treatment processes, the highest removal rate was observed for Ibuprofen and the lowest for Diclofenac, 92% and 26%, respectively. Nakada and coworkers (2006) confirm the good removal efficiency of Ibuprofen (>90%), and the lower efficiency (<50%) for Ketoprofen and Naproxen.

In contrast, Caffeine is efficiently removed in well functioning WWTPs (>99%). Nevertheless, it is ubiquitously present in the aqueous environment, due to its extensive use (Buerge et al., 2003). It degrades relatively slowly by direct photolysis (>170 h in artificial sunlight), but enhanced photodegradation was observed in waters containing fulvic acids (Jacobs et al., 2012).

Hollender and co-workers (2009) studied in a typical Swiss municipal WWTP (Wüeri in Regensdorf) the removal efficiency for 220 micropollutants by conventional secondary treatment, and upgraded with post-ozonation followed by sand filtration. The secondary treatment was very effective for easily biodegradable compounds such as Ibuprofen (eliminations >85%). Compounds known to be moderately persistent such as Diclofenac, Naproxen, Bezafibrate, sulfonamide and macrolide antibiotics, several beta blockers, and the anticorrosive agents Benzotriazole and 5-Methylbenzotriazole showed eliminations between 20 and 80%. As expected, Carbamazepine and a few triazine derivatives almost completely persisted during activated sludge treatment. The comparison of the Fate Sees data with the secondary effluent concentrations of Hollender et al. (2009) shows a relatively good agreement (average concentration levels; Table 23) (see also for pesticides under 6.3.4).

Table 23: Comparison of the Fate Sees data with the WWTP Regensdorf (Switzerland); secondary effluent concentrations in ng/l (Hollender et al., 2009).

	WWTP Regensdorf (CH)	Fate Sees
	Average levels	Average levels
Bezafibrate	55-67	25
Clindamycine	18-64	70
Clofibric acid	n.d.-44	5
Fluconazole	n.d.-58	108
Ketoprofen	98-160	86
Trimethoprim	76-111	229
Venlafaxine	150-270	119

Gracia-Lor and co-workers (2010) analysed the 20 most consumed pharmaceuticals of different therapeutic classes in Spain in urban wastewater and surface water samples. The optimized method was applied to the analysis of 42 urban WWTP influent and 42 effluent water samples (Castellón province), with the result that 17 out of 20 investigated compounds were detected in the samples. Analgesics and anti-inflammatories (NSAIDs), cholesterol lowering drugs and lipid regulators were the major groups found, with Diclofenac, Ketoprofen, Naproxen, 4-Aminoantipyrine, Bezafibrate, Gemfibrozil and Venlafaxine being the most frequently detected. The highest concentrations in influents were found for Acetaminophen, Salicylic acid and Ibuprofen. Diclofenac, Naproxen, Ketoprofen, Gemfibrozil, Bezafibrate, and 4-Aminoantipyrine were present in all influent and effluent samples analyzed at concentration levels normally in the range of high ng/l or low µg/l. Table 24 compares the maximum and median levels found in WWTPs in the Castellón province (Spain) with the Fate Sees data, and shows that the results for the maximum levels agree relatively well (with the exception of Ibuprofen, and some antibiotics). The median concentrations of Fate Sees are however in most cases much lower than in the study by Gracia-Lor et al. (2010, 2012). In a second monitoring campaign additional compounds including many antibiotics were analysed (Gracia-Lor et al., 2012). This comparison shows that it is difficult to compare results of median concentrations of one WWTP (or a small number) with the median of Fates Sees (n=90).

Table 24: Comparison of the Fate Sees data with three urban WWTPs in the Castellón province (Spain); effluent concentrations in (µg/l); 84 samples analysed (Gracia-Lor et al., 2010, 2012).

	Castellón province (Spain)		Fate Sees	
	Maximum levels	Median conc.	Maximum levels	Median conc.
Diclofenac	0.74	0.33	0.17	0.043
Ibuprofen	n.d.	n.d.	2.13	0.007
Ketoprofen	0.62	0.30	1.65	0.000
Naproxen	0.72	0.17	0.96	0.008
Atorvastatin	0.16	0.02	0.073	0.000
Bezafibrate	0.39	0.07	0.39	0.004
Gemfibrozil	1.24	0.54	3.62	0.005
Paroxetine	n.d.	n.d.	n.d.	0.000

Venlafaxin	0.30	0.14	0.55	0.097
Risperidone	n.d.	n.d.	0.086	0.003
Alprazolam	n.d.	n.d.	0.033	0.000
Ciprofloxacin	1.08	0.70	0.264	0.082
Enrofloxacin	n.d.	n.d.	n.d.	n.d.
Flumequine	n.d.	n.d.	0.026	n.d.
Sulfamethoxazole	0.06	0.05	1.147	0.068
Sulfadiazine	n.d.	n.d.	0.105	n.d.
Lincomycine	0.16	0.01	0.317	0.000
Clindamycine	0.02	0.02	0.277	0.046
Trimethoprim	0.10	0.09	0.800	0.178

In a long term study by [Gros and co-workers \(2010\)](#), which covered 4 sampling periods over three years, a total number of 84 samples, specifically 28 influents, effluents, from seven WWTPs located in the main cities along the Ebro river Basin (North East of Spain), as well as receiving river waters, were analyzed to assess the occurrence of 73 pharmaceuticals covering several medicinal classes. The maximum effluent concentrations are compared in Table 25 with the results of Fate Sees. There is a relatively good agreement for most compounds, with the exception of Diclofenac, Carbamazepine, and Ranitidine.

Table 25: Comparison of the Fate Sees data with 7 WWTPs located in the main cities along the Ebro river basin (Spain); maximum effluent and river water concentrations in (ng/l) ([Gros et al., 2010](#)).

Compounds	WWTP effluents	River waters	Fate Sees
Naproxen	1740	165	958
Diclofenac	1090	120	174
Ibuprofen	2400	185	2128
Ketoprofen	2980	< LOQ	1653
Codeine	825	213	826
Bezafibrate	454	16	344
Clofibric acid	30	104	127
Gemfibrozil	520	97	3619
Atorvastatin	155	34	73
Fluoxetine	21	410	22
Paroxetine	14	2	n.d.
Carbamazepine	366	98	4609
Ciprofloxacin	313	109	265
Sulfamethoxazole	620	36	1147
Trimethoprim	463	114	800
Ranitidine	1452	115	44

The occurrence and degradation of 32 emergent contaminants by AOPs in the municipal WWTP of Vidy at the city of Lausanne was investigated by [De la Cruz et al. \(2012\)](#). This WWTP treats water of 180,000 inhabitants and several hospitals, employing a treatment based on biological activated sludge. Table 26 gives the concentrations of the contaminants after the secondary treatment. The comparison to the Fate Sees data shows

a relatively good agreement for most of the substances, with the exception of only Bezafibrate, and Diclofenac.

Table 26: Comparison of the Fate Sees data with the WWTP Vidy at the city of Lausanne (Switzerland); effluent concentrations of secondary treatment in ng/l (De la Cruz et al., 2012).

	WWTP Lausanne (CH)	Fate Sees
	Average levels	Average levels
Bezafibrate	426	25
Gemfibrozil	25	138
Carbamazepine	263	832
Diclofenac	518	50
Ibuprofen	112	81
Ketoprofen	123	86
Naproxen	178	27
Ciprofloxacin	129	96
Sulfamethoxazole	578	142
Trimethoprim	131	229

Rosal et al. (2010) performed a one-year survey of the WWTP of Alcalá de Henares (Madrid), which treats a mixture of domestic and industrial wastewater, and studied the occurrence and fate of 84 pollutants (mainly PPCPs). This plant is a “conventional” activated sludge WWTP with a biological nutrient removal process aimed to the elimination of phosphorous and nitrogen. The following Table 27 compares the maximum and average concentration levels of the effluent with the Fate Sees data. This comparison shows that the maximum concentrations of the Fate Sees data are for most compounds similar to the data of Rosal et al. (2010) (exceptions: Carbamazepine, Ciprofloxacin, Diuron, Fluoxetine, Galaxolid, Ranitidine). The average levels however are in the same range only for Bezafibrate, Codeine, Diclofenac, Diuron, Ibuprofen, Ketoprofen, Sulfamethoxazole, Triclosan, and Trimethoprim.

Table 27: Comparison of the Fate Sees data with a WWTP from Spain (effluents); concentrations in (ng/l) (Rosal et al., 2010).

	WWTP Alcalá de Henares (Spain)			Fate Sees	
(ng/l)	Maximum	Average	Removal efficiency (%)	Maximum	Average
Bezafibrate	280	128	9	344	25
Caffeine	1589	1176	95	3002	191
Carbamazepine	173	117	10	4609	832
Ciprofloxacin	5692	2378	57	265	96
Codeine	319	160	69	826	71
Diclofenac	431	220	5	174	50
Diuron	81	42	62	1426	62
Fluoxetine	929	223	62	22	2
Gemfibrozil	5233	845	76	3619	138
Ibuprofen	653	135	95	2129	81
Ketoprofen	539	392	11	1653	86

Naproxen	2208	923	61	958	27
Ranitidine	942	360	31	44	7
Sulfamethoxazole	370	231	17	1147	142
Trimethoprim	148	99	5	800	229

A five-month monitoring program was undertaken in South Wales in the UK to determine the fate of 55 PPCPs, endocrine disruptors and illicit drugs in two contrasting wastewater plants utilizing two different wastewater treatment technologies: activated sludge and trickling filter beds (Kasprzyk-Hordern et al., 2009). The efficiency of the removal of PPCPs was found to be strongly dependent on the technology implemented in the WWTP. In general, the WWTP utilizing trickling filter beds resulted in, on average, less than 70% removal of all 55 PPCPs studied, while the WWTP utilizing activated sludge treatment gave a much higher removal efficiency of over 85%. Also here a comparison shows that the median concentrations of Fate Sees are in most cases much lower (Table 28). Exceptions are Diclofenac, Carbamazepine, and Sulfamethoxazole. The maximum concentrations, however, are relatively similar for several pharmaceuticals (Trimethoprim, Ibuprofen, Diclofenac, Naproxen, Carbamazepine, Clofibric acid, and Bezafibrate).

Table 28: Comparison of the Fate Sees data with two English WWTPs (effluents); concentrations in (ng/l) (Kasprzyk-Hordern et al., 2009).

	South Wales (UK)		Fate Sees	
(ng/l)	Range	Median	Range	Median
Trimethoprim	625-3052	1152	11-800	178
(WWTP 2)	385-1218	876		
Ibuprofen	131-424	263	0-2128	7
(WWTP 2)	65-491	143		
Diclofenac	33-142	98	0-174	43
(WWTP 2)	6-496	179		
Ketoprofen	<3-33	16	0-1653	0
(WWTP 2)	7-37	18		
Naproxen	234-703	370	0-958	8
(WWTP 2)	<2-269	170		
Codeine	2940-15593	5271	0-826	21
(WWTP 2)	1457-4178	2716		
Tramadol	24132-97616	43813	0-1166	218
(WWTP 2)	12779-56810	28147		
Carbamazepine	644-4596	2499	0-4609	752
(WWTP 2)	152-2324	826		
Sulfamethoxazole	<3-23	10	0-1147	68
(WWTP 2)	4-44	19		
Clofibric acid	<1-75	15	0-127	0
(WWTP 2)	<1-48	6		
Bezafibrate	<85-667	231	0-344	3,5
(WWTP 2)	<94-393	177		
Ranitidine	<9-455	224	0-44	0
(WWTP 2)	15-783	425		

Diltiazem	95-642	267	0-64	6,4
(WWTP 2)	108-1156	357		

Martínez Bueno et al. (2012) carried out an almost two-years monitoring program in five municipal WWTPs located in the north, centre and south-east of Spain. The study evaluated the occurrence and persistence of a group of 100 organic compounds belonging to several chemical groups (PPCPs, pesticides and metabolites). The average removal efficiencies of the WWTPs varied from 20% (Erythromycin) to 99% (Acetaminophen). Mean concentrations detected for the analytes identified ranged from 7 ng/l to 59 µg/l in influent samples and from 5 ng/l to 32 µg/l in effluents. Given this wide range of concentrations, it is noteworthy that a group of only 20 compounds was responsible for 83% of the total load of pollutants in the effluent. This group included 13 pharmaceuticals, mainly analgesics/anti-inflammatories (Diclofenac, Codeine, Naproxen, Ibuprofen), antibiotics (Ofloxacin, Ciprofloxacin, Erythromycin), diuretics (Furosemide, Hydrochlorothiazide), lipid regulators (Gemfibrozil), b-blockers (Atenolol), ulcer healings (Ranitidine) and the stimulant Caffeine. It is also remarkable the presence at very high concentrations of a group of metabolites such as Fenofibric acid (metabolite of Fenofibrate), Paraxanthine (metabolite of Caffeine) and four of the main metabolites of the antipyretic drug Dypirone (Martínez Bueno et al., 2012). These results are listed in Table 29 together with the Fate Sees data. The average levels are in good agreement to the results of Fate Sees only for about 50% of the pharmaceuticals (Citalopram, Codeine, Ibuprofen, Ketoprofen, Lincomycin, Sulfadiazine, Sulfamethoxazole, Trimethoprim, and Venlafaxine).

Table 29: Comparison of the Fate Sees data (average) with 5 Spanish WWTPs; mean concentrations of an almost two years monitoring study; samples were taken monthly; effluent concentrations in (ng/l) (Martínez Bueno et al., 2012).

	Alemeria (n=15)	Cantabria (n=8)	Madrid 1 (n=22)	Madrid 2 (n=12)	Barcelona (n=9)	Average	Median	Fate Sees (average)
Bezafibrate	174	217	156	300	258	221	217	25
Caffeine	5521	1531	882	577	215	1745	882	191
Carbamazepine	274	85	115	154	254	176	154	832
Ciprofloxacin	712	94	1918	1526	309	912	712	96
Citalopram	176	51	53	0	151	86	53	34
Clofibrac acid	95	0	238	14	102	90	95	5
Codeine	180	0	133	107	18	88	107	71
Diclofenac	249	76	5703	13814	0	3968	249	50
Fluoxetine	230	0	259	208	14	142	208	2
Gemfibrozil	6805	4015	1029	2191	5475	3903	4015	138
Ibuprofen	1181	0	54	0	0	247	0	81
Ketoprofen	12	68	50	0	62	38	50	86
Lincomycin	140	0	14	0	96	50	14	31
Naproxen	5433	498	1118	1046	84	1636	1046	27
Paroxetine	381	0	30	0	0	82	0	0
Ranitidine	873	222	614	903	543	631	614	7
Sulfadiazine	0	0	0	0	19	4	0	4
Sulfamethoxazole	548	246	208	257	328	317	257	280
Trimethoprim	371	129	118	196	193	201	193	229
Venlafaxine	324	273	189	0	448	247	273	119

Miége et al. (2009) collected data on PPCPs in WWTPs from 117 scientific publications in a data-base. A comparison with the Fate Sees data is shown in Table 30. Miége and co-workers report much higher median (and maximum) values for Diclofenac, Ibuprofen, Ketoprofen, Naproxen, Bezafibrate, Gemfibrozil, Clofibric acid, Bisopropol, and Triclosan. In contrary, relatively similar concentrations are found for the substances Sulfamethoxazole, Trimethoprim, Carbamazepine, and Ciprofloxacin (and the maximum for Ketoprofen). A reason for the lower median pharmaceutical concentrations of the Fate Sees study might be that also several industrial WWTPs were included in the study.

Table 30: Comparison of the Fate Sees data with WWTP effluent data from a literature data base; concentrations in (µg/l) (Miége et al., 2009).

(µg/l)	Miége et al. (2009)				Fate Sees			
	Median	Max.	n	Freq.(%)	Median	Max.	n	Freq.(%)
Diclofenac	0,42	1,95	101	85	0,043	0,17	90	90
Ibuprofen	0,80	24,6	109	93	0,007	2,13	90	56
Ketoprofen	0,21	1,62	53	73	0,000	1,65	90	71
Naproxen	0,88	33,9	53	87	0,008	0,96	90	64
Bezafibrate	0,25	4,80	21	78	0,004	0,34	90	62
Gemfibrozil	0,60	1,34	21	70	0,005	3,62	90	59
Clofibric acid	0,15	0,23	24	55	0,000	0,13	90	26
Sulfamethoxazole	0,07	0,32	11	73	0,068	1,15	90	81
Trimethoprim	0,06	0,55	27	93	0,178	0,80	30	93
Carbamazepine	0,52	2,30	63	100	0,752	4,60	90	89
Clotrimazole	0,01	0,03	3	100	0,000	0,01	90	33
Tamoxifen	0,20	0,37	3	19	0,000	0,01	90	2
Bisopropol	0,64	1,43	18	100	0,016	0,42	90	97
Triclosan	0,13	0,43	19	100	0,000	4,26	90	41
Ciprofloxacin	0,07	0,14	29	91	0,082	0,27	30	90

Moreover, a good one-year WWTP monitoring study to evaluate the occurrence, persistence and fate of a group of 14 organic compounds in a WWTP located in the south of Spain was published by Gómez and co-workers (2007). Diclofenac concentrations ranged between 0.14-2.2 µg/l with a median of 0.9 µg/l. Table 31 shows that a comparison of these data from one WWTP with the Fate Sees data is very difficult.

Table 31: Comparison of the Fate Sees data with a one-year WWTP study (effluent) in south Spain; concentrations in (µg/l) (Gómez et al., 2007).

	South Spain		Fate Sees	
	Range	Median	Range	Median
Ibuprofen	0.24-28	7.1	0-2.1	0.007
Caffeine	1.4-44	12	0-3.0	0.035
Triclosan	0.08-0.40	0.2	0-4.3	0.000
Diclofenac	0.14-2.2	0.9	0-0.174	0.043
Carbamazepine	0.11-0.23	0.13	0-4.6	0.752

Codeine	0.9-8.1	3.7	0-0.826	0.021
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Clara and co-workers (2005) investigated several Austrian WWTPs for the occurrence and removal of PPCPs and endocrine disrupting compounds. Table 32 reports the range of concentration levels found in the effluents of these WWTPs. Also here, it is obvious that a comparison of median concentrations with the Fate Sees data is difficult; however, for Carbamazepine and Sulfamethoxazole, the agreement is good.

Table 32: Comparison of the Fate Sees data with Austrian WWTPs; effluent concentrations of secondary treatment in (ng/l) (Clara et al., 2005).

	WWTPs in Austria	Fate Sees
	Mean levels	Median levels
Diclofenac	780-1680	43
Ibuprofen	20-2400	7
Bezafibrate	n.d.-4800	4
Carbamazepine	465-1594	752
Sulfamethoxazole	n.d.-91	68

Vieno and coworkers (2007) studied twelve WWTPs in Finland, where the fluoroquinolone antibiotics Ciprofloxacin, Norfloxacin, and Ofloxacin were eliminated by >80%, and the β -blockers Acebutolol, Atenolol, Metoprolol and Sotalol in average by less than 65%; the elimination varied greatly between the treatment plants. The concentrations for Carbamazepine and Ciprofloxacin are in good agreement to Fate Sees data (Table 33).

Table 33: Comparison of the Fate Sees data with WWTP effluents from Finland (ng/l); (Vieno et al., 2007).

	WWTPs in Finland		Fate Sees	
	Median	Maximum	Median	Maximum
Carbamazepine	500	2440	752	4609
Ciprofloxacin	70	130	82	265

Diclofenac

Zhang and co-workers (2008) review the world-wide occurrence of Diclofenac (and Carbamazepine) in WWTP effluents. Concentrations for Diclofenac range from ca. 100-1500 ng/l with an approximate median value of 300 ng/l. Moreover, some information on Diclofenac in WWTP influents and effluents is available from the KNAPPE project (Knappe, 2008), presented in Table 34.

Table 34: Concentrations of Diclofenac in WWTP effluents ($\mu\text{g/l}$). Data from the Knappe Project.

	Median	Maximum	Number of samples
Germany	1.34	10.0	382
France	0.31	0.92	46
Spain	0.31	2.20	47

UK	0.34	2.30	15
Austria	1.84	3.00	7
Greece	0.07	0.89	6
Italy	0.68	5.45	8
Average	1.29	4.59	

Table 35 summarizes in comparison to Fate Sees some monitoring data on Diclofenac.

Table 35: Concentrations of Diclofenac in WWTP effluents (µg/l).

Country	City or Region	Concentration	Reference
Europe		Median: 0.043 Average: 0.050 Max.: 0.174	Fate Sees
Germany		90 th percentile: 1.60	Ternes (1998)
EU median	5 countries	Median: 0.29	Paxéus (2004)
Spain	South	Median: 0.90	Gómez et al. (2007)
EU median		Median: 0.42 Max.: 1.95	Miége et al. (2009)
UK	South Wales	Median: 0.14	Kasprzyk-Hordern et al. (2009)
Switzerland	Regensdorf	Average: 0.69 (influent: 1.45)	Singer et al. (2010)
Spain	Castellón	Median: 0.33 Max.: 0.74	Gracia-Lor et al. (2010,2012)
Spain	Ebro river region	Max.: 1.09	Gros et al. (2010)
Spain	Alcalá de Henares	Max.: 0.43 Average: 0.22	Rosal et al. (2010)
Switzerland	Lausanne	Average: 0.52	De la Cruz et al. (2012)

In comparison to the effluents data for Diclofenac from the literature presented in the Table 34 and Table 35, in our EU-wide survey (Fate Sees) lower Diclofenac concentrations were found (with the exception of Kasprzyk-Hordern et al. (2009) (median: 0.14 ng/l), and Rosal et al. (2010) (average: 0.22 ng/l). The maximum concentration of Fate Sees was 174 ng/l, median 43 ng/l, average 50 ng/l, and 90th percentile 103 ng/l. Therefore, the quantification of Diclofenac with the help of the internal standard Ibuprofen ¹³C₃ was double-checked, but no quantification error could be found. Chromatograms for Diclofenac are shown in the Annex. The reason for these relatively low Diclofenac concentrations found in the Fate Sees study remains therefore unresolved. The JRC-IES laboratory participated in two interlaboratory studies on non-steroidal anti-inflammatory drugs (NSAIDs) (Farré et al., 2008; Heath et al., 2010). Also here, relatively high deviations were observed for the laboratories, and the JRC-IES laboratory results were below the median. It must be concluded that the concentrations found for Diclofenac in WWTP effluents in Fate Sees are lower than in other studies. A reason might be problems with different analytical standards, as noticed during the interlaboratory studies. However, also for other substances a comparison with other studies is difficult. There is also a great discrepancy in literature data for the removal of Diclofenac in WWTPs (i.e., 0–69%) (Ternes, 1998; Radjenović et al., 2007; 2009; Lindqvist et al., 2005).

Triclosan

Triclosan (2,4,4'-trichloro-2'-hydroxydiphenylether) is currently used as an antimicrobial agent in toothpaste, mouthwash, liquid soap and in functional clothing such as functional shoes and underwear, as a stabilizing agent in a multitude of detergents and cosmetics and as an antimicrobial agent in polymeric food cutting boards. The primary emission route for Triclosan after usage is through wastewater. In WWTPs ~90% of the incoming Triclosan is removed from the water (also by adsorption to sludge) (Bester, 2003; Heidler and Halden, 2008; Singer et al., 2002), which is a high but not complete removal. As a result, it has been found in WWTP effluents as well as in surface water and ground water (and sewage sludge) in many countries (Chen et al., 2012b).

Table 36 gives some examples of European WWTP effluent concentrations for Triclosan, which agree well with Fate Sees. In the Fate Sees study a quite high maximum concentration level of 4.3 µg/l was found. It must be noted that the real Triclosan levels of Fate Sees are probably the double of the reported concentrations because the used HDPE bottles are not recommended for use when sampling for this compound (due to adsorption) (Vanderford et al., 2012, chapter 2, page 19).

Table 36: Comparison of the Fate Sees data for Triclosan with other studies; WWTP effluent concentrations in (ng/l).

Location	(ng/l)	Reference
Fate Sees (Europe; n=90)	Max.: 4259 Average: 75	This report
WWTPs around Lake Greifensee (CH)	Range: 42-213	Singer et al. (2002)
WWTPs (CH)	Range: 70-650	Lindström et al. (2002)
WWTP Dortmund (Germany)	Range: 43-59	Bester (2003)
WWTP in south Spain	Range: 80-400 Median: 200	Gómez et al. (2007)
WWTPs in Europe (n=19)	Max.: 430 Median: 130	Miége et al. (2009)
WWTP Alcalá de Henares (Spain)	Max.: 512 Average: 219	Rosal et al. (2010)
WWTP Vidy Lausanne (CH)	Average: 135	De la Cruz et al. (2012)
5 WWTPs (Spain)	Max.: 3678 Average: 301	Martínez Bueno et al. (2012)

6.3.4. Pesticides

In comparison to PPCPs, less information is available on the occurrence of pesticides in WWTPs, because direct inputs via agricultural field run-off might be more important for pesticides exposure. The average DEET concentration found in Fate Sees (678 ng/l) is in very good agreement to the Swiss Micropoll Project (593 ng/l) (see before) (Micropoll, 2011; 2012). Clara and co-workers (2012) monitored over a one-year period several priority compounds of the WFD (including the pesticide Diuron) in nine Austrian WWTPs. The median concentration of 60 measurements was 40 ng/l, slightly higher than

the Fate Sees median of 12 ng/l. Table 37 shows also good match for most other pesticides (for the average levels).

Table 37: Comparison of the Fate Sees data for some pesticides with other studies; WWTP effluent average concentrations in (ng/l).

Country	CH	CH	CH	Spain	Austria	Europe
Location	Regensdorf	Regensdorf	Lausanne	5 WWTPs	9 WWTPs	
Reference	Hollender et al. (2009)	Singer et al.(2010)	De la Cruz et al. (2012)	Martinez Bueno et al.(2012)	Clara et al. (2012)	Fate Sees
Atrazine		30	9	64		4
Diuron	n.d.-70	40	57	259	40	62
DEET	360-600					678
Desethylatrazine	36-50	30				14
Diazinon	59-492	40				21
Isoproturon	38-159	30	< 10	127		10
Linuron	21-110					40
MCPA	n.d.-22					150
Mecoprop	163-3488	1010	34			127
Simazine				44		26
Terbutylazine		10				91
Sulfamethoxazole		140				142

6.3.5. Sweeteners Acesulfame K and Sucralose

Artificial low-calorie sweeteners are consumed in considerable quantities with food and beverages. After ingestion, some sweeteners pass through the human metabolism largely unaffected, are quantitatively excreted via urine and feces, and thus reach the environment associated with domestic wastewater. Acesulfame K and Sucralose were recently identified as ubiquitous (emerging) environmental contaminants. They were detected in untreated and treated wastewater (Buerge et al., 2009; Ferrer and Thurman, 2010; Scheurer et al., 2009), in surface waters (Buerge et al., 2009; Ferrer and Thurman, 2010; Loos et al., 2009b; Scheurer et al., 2009), groundwater (Buerge et al., 2009; Ferrer and Thurman, 2010), ocean waters (Mead et al., 2009), and even in tap and drinking water samples (up to 2.6 µg/l for Acesulfame K and 2.4 µg/l for sucralose) (Buerge et al., 2009; Mawhinney et al., 2011).

Concentrations of Acesulfame K and Sucralose in WWTP influents and effluents are quite similar; in Switzerland between 12-46 µg/l for Acesulfame K and between 2-9 µg/l for Sucralose were found (Hollender et al., 2009; Scheurer et al., 2009). These sweeteners are not eliminated in WWTPs and are quite persistent in surface waters, and in soil aquifer treatment. Due to these properties, Acesulfame K and Sucralose are ideal markers for the detection of domestic wastewater in natural waters, particularly groundwater (Buerge et al., 2009; Oppenheimer et al., 2011; Scheurer et al., 2009; 2010).

In our EU-wide study we found maximum concentration levels of around 2.5 mg/l Acesulfame K and 13 µg/l Sucralose, respectively. The median levels were (in good

agreement to literature data; see above) 14.3 µg/l and 1.7 µg/l for Acesulfame K and Sucralose, respectively. These sweeteners were not detected in all WWTPs because also some industrial plants were included in our study. Hence, Acesulfame K was the substance with the highest overall concentrations found in the Fate Sees study.

Note that for the quantification of Acesulfame K a recovery correction factor of 10 was applied, because it had a very low SPE recovery of only around 10 %.

6.3.6. Organophosphate ester flame retardants

Organophosphate esters are high-production-volume chemicals used as flame retardants and plasticizers to protect or to enhance the properties of plastics, textiles, furniture and many other materials. The widespread usage, which may even increase due to the increasing global ban of brominated diphenylethers as flame retardants, and the diffusion from host materials result in continuous release of Organophosphate esters and their distribution through water, especially wastewater, and air, particularly associated with airborne particulate matter (Reemtsma, et al., 2008).

Nowadays, TCPP is more prominent in effluents than TCEP, reflecting the phasing out of the latter. Chlorinated organophosphate esters flame retardants are hardly removed in WWTPs. Of a series of 29 polar trace pollutants, TCPP was among the 10 with no significant elimination in WWTP, whereas TCEP showed slight removal (only 20%) (Reemtsma, et al., 2006). This European WWTP survey by Reemtsma and co-workers confirmed that TCEP and TCPP are routinely detected in WWTP effluents, typically at concentrations of a few hundred ng/l. TCPP and TCEP were determined with mean concentrations of 0.6 and 0.2 µg/l, respectively. These results were confirmed in effluents of 16 WWTPs in Austria, where median concentrations for TCPP, TCEP, and TBEP were 580 ng/l, 74 ng/l, and 130 ng/l, respectively (Martínez-Carballo et al., 2007).

These values are in good agreement to the Fate Sees study where median levels were: TCPP 620 ng/l, TCEP 71 ng/l, and TBEP 190 ng/l.

6.3.7. Fragrances / musk compounds

Synthetic polycyclic musks are extensively used as fragrance ingredients in a number of consumer and personal care products, such as in laundry detergents, fabric softeners, cleaning agents, perfumes, shampoos, soaps, deodorants, detergents, body lotions, etc. Their lipophilic characteristics and degradation rates are similar to some well known hydrophobic and semi-volatile organic pollutants. They have been detected in various environmental samples worldwide, including in air, freshwater, sewage sludge, sediments, aquatic biota, and even in human adipose tissue, breast milk and blood samples. Tonalide® (AHTN) and Galaxolide® (HHCB) are the two largest volume products in the group of polycyclic musks, representing about 95% of the EU market. Major sources of polycyclic musks are households. In WWTPs overall removal efficiencies between approx. 50% and more than 95% are observed during biological

wastewater treatment and are mainly removed by sorption onto sludge (Clara et al., 2011; Joss et al, 2005; Kupper et al., 2006; Wu and Ding, 2010).

Reemtsma and co-workers (2006) investigated four musk components in their WWTP study (Galaxolide® (HHCB), Tonalide® (AHTN), Muskketone and Muskxylene); a median total concentration of 0.55 µg/l was found, and Galaxolide® (HHCB) and Tonalide® (AHTN) prevailed, whereas Muskketone and Muskxylene were hardly detected at measurable quantities. In our study (Fate Sees), however, Galaxolid® and Tonalide® were not detected frequently (detection frequency: 22 % and 1 %, respectively). Cashmeran® was found more often (57 %). The maximum concentrations found for Galaxolid®, Tonalide®, and Cashmeran® were 260 ng/l, 77 ng/l, and 480 ng/l, respectively. Table 38 compares the Fate Sees data of HHCB and AHTN with other WWTP studies. Much higher concentrations were found in other studies.

Table 38: Comparison of the Fate Sees data for HHCB and AHTN in WWTP effluents (ng/l).

	HHCB (Galaxolide®)	AHTN (Tonalide®)	Reference
Europe	Max.: 260 Average: 21 Median: 0,000 (detection freq. <50%)	Max.: 77 Average: 1 Median: 0,000 (detection freq. <50%)	Fate Sees
WWTPs in Austria	Mean: 451-870	Mean: 144-170	Clara et al. (2005)
WWTP Mittleres Emmental, Hasle (CH)	Mean: 770	Mean: 320	Kupper et al. (2006)
Literature data base (worldwide)	Max.: 1080 Median: 600 (n=9)	Max.: 200 Median: 160 (n=8)	Miége et al. (2009)
WWTP Alcalá de Henares (Spain)	Max.: 2766 Average: 1225 Removal efficiency: 88%	Max.: 315 Average: 146 Removal efficiency: 85%	Rosal et al. (2010)
WWTPs in Spain	Max.: 25.9 µg/l Average: 4125 Median: 3365	Max.: 2.1 µg/l Average: 524 Median: 337	Martinez Bueno et al. (2012)

6.3.8. X-ray contrast agents

X-ray contrast agents are only found in some WWTP effluents (detection frequency between 15-47 %), because they are mainly coming from hospital effluents. Very high single maximum concentrations were found in Fate Sees (e.g.: 150 µg/l for Iopromid in a WWTP from Portugal).

Table 39 compares the Fate sees data with literature results. Average concentrations of Fate Sees are in very good agreement to the Micropoll Project in Switzerland (Micropoll, 2011; 2012).

Table 39: Comparison of the Fate Sees data for x-ray contrast agents with literature data; WWTP effluent concentrations in (ng/l).

	Amidotrizoic acid	Iomeprol	Iopamidol	Iopromid	Iohexol	Reference
Europe	Max.: 8400 Average: 619 Median: 0,00	Max.: 12000 Average: 376 Median: 0,00	Max.: 6100 Average: 144 Median: 0,00	Max.: 150000 Average: 2680 Median: 0,00	Max.: 7700 Average: 158 Median: 0,00	Fate Sees
Switzerland	Average:598	Average: 380	Average: 377	Average: 876		Micropoll, 2011
Regensdorf (CH)			Average:31		Average:31	Hollender et al., 2009
Lausanne (CH)			Average:1716	Average: < 600		De la Cruz et al., 2012
Austria				Mean: n.d.-5060		Clara et al., 2005

6.3.9. Trace elements measured by ICP-AES (JRC-IES)

The trace elements Hg (EQS: 0.05 µg/l), Ag, Al, As, Ba, Be, Cd (EQS: 0.08-0.25 µg/l), Co, Cr, Cu, Mg, Mn, Mo, Ni (EQS: 2 µg/l), Pb (EQS: 1.2 µg/l), Sb, Se, Ti, and Zn were measured in all 90 effluent samples by ICP-AES (Annex 10).

Magnesium (Mg), Manganese (Mn), and Zinc (Zn) were regularly detected in the microgram to milligram per liter concentration range. The priority substances of the WFD Mercury (Hg), and Cadmium (Cd) were in all cases below the LOD of 1 µg/l. The EQS value for Nickel (2 µg/l) was exceeded in three water samples (50-420 µg/l) (samples 150, 151, 152).

6.4. Bioanalytical results

6.4.1. Estrogenicity

From the total number of 75 analyzed WWTP effluents, 27 sample extracts showed estrogenic activity higher than the detection limit > 0.5 ng/l EEQ (estrogen equivalents) (see Annex 9). Estrogenic activity expressed as estradiol equivalents varied (in positive samples) from 0.53 to 17.9 ng/l EEQ. Median and arithmetic mean of all tested samples were < 0.5 ng/l and 0.9 ng/l EEQ, respectively (Table 40).

The levels of detected EEQs are well comparable to the results of previous studies evaluating estrogenic activity of European WWTP effluent samples by different *in vitro* bioassays. For example, [Svenson et al. \(2003\)](#) used human estrogen receptor, hosted in a yeast strain, to quantify estrogenicity in samples of effluents from 20 Swedish municipal WWTPs. The treatment plants were selected to represent different treatment processes regarding chemical precipitation (coagulation and precipitation by Al or Fe to remove phosphorus and coagulate dissolved organic material) and microbial procedures. The discharge from Swedish domestic WWTPs contained estrogenic compounds at levels ranging $< 0.1 - 15$ ng/l EEQ (Table 40). The other larger studies evaluating estrogenicity of European WWTPs effluents were for example those performed by [Korner et al. \(2001\)](#), [Vethaak et al. \(2005\)](#), [Aerni et al. \(2004\)](#) or [Cargouet et al. \(2004\)](#). After the exclusion of one outlying value reported by [Aerni et al. \(2004\)](#), the levels of measured EEQ in all these studies varied from less than 0.03 to 24 ng/l, which is also in good agreement with the results present in this report. However, all these studies reported higher frequency of detection of positive samples in comparison to our study. Several reasons could be considered. First, to provide reliable results for relatively large number of samples investigated in the present study, we have done no further concentrations of the initially negative samples. This is the reason why our detection limit was higher in comparison to previously reported studies, and resulted in decreased frequency of positive "estrogenic" samples. Second, higher levels of EEQ in previously published studies were often detected at municipal WWTPs with other treatment steps than activated sludge with nitrification (e.g. [Svenson et al., 2003](#)) while this type of WWTP was the most frequent in our study. Finally, 9 out of the 75 sample extracts were either highly cytotoxic or elicited anti-estrogenic effects, which might mask effects of estrogens in complex mixtures (see Table 40).

Table 40: Comparison of the FATE SEES data on estrogenic activity with available literature data.

Location	Type of waste water	Type of treatment	Median EEQ (ng/l)	Average EEQ (ng/l)	Max. EEQ (ng/l)	LOD EEQ (ng/l)	n WWTPs	Freq. (%)	Reference
EU wide study	M, I	Different treatment steps but mostly activated sludge with (de)nitrification	<0.5	0,9	17,9	0,5	75	36	FATE SEES study
Sweden	M	Different treatment steps, only 2 WWTP activated sludge with nitrification	2,7	4,9	14,9	0,1	20	88	Svenson et al. 2003
Southwestern Germany	M	Different treatment steps but mostly activated sludge with (de)nitrification	1,6	2,26	7,8	0,03	16	97	Korner et al., 2001
The Netherlands	M	Different treatment steps	0,3	n.a.	2,2	0,1	10	90	Vethaak et al., 2005
Switzerland	M	Activated sludge with (de)nitrification	2.1 -5.3	n.a.	53,0	n.a.	5	90	Aerni et al., 2004
France Paris area	M	Activated sludge, activated sludge with (de)nitrification, biofilters	11	12	24,0	n.a.	4	100	Cargouet et al., 2004
The Netherlands	I	n.a.	0,9	n.a.	9,5	n.a.	3	100	Vethaak et al., 2005
Southwestern Germany	I	Activated sludge with (de)nitrification, trickling filter	<0.03- 0.65	- 0,325	0,7	0,03	2	50	Korner et al., 2001

M – Municipal (mainly domestic waste waters, industrial waters may contribute only partially)

I – Industrial

n.a. – information was not available in the referenced paper

6.4.2. Dioxin-like activity

To evaluate approximate levels of dioxin-like activity, we studied 25 WWTP effluent sample extracts. Evaluated samples were those that were first delivered to our laboratories at RECETOX, Masaryk University, Brno; no other selection criteria were used. Twenty one out of the 25 tested sample extracts exceeded the detection limit (0.10 ng/l TEQ_{bio}) but the maximum detected dioxin-like activity was relatively low reaching only 0.44 ng/l TEQ_{bio} with median around 0.15 ng/l TEQ_{bio} (Table 41).

To our knowledge, only a limited number of papers reported about dioxin-like activity in dissolved water phase. Most of the known ligands for aryl hydrocarbon receptor, which is responsible for dioxin-like effects, are fairly hydrophobic compounds, and their presence in other matrices (e.g. particulate matter) is more likely. Nevertheless, dioxin-like activity has been reported also in water samples in some previous studies. For example Reungoat et al. (2010) tested dioxin-like activity of final effluent of Australian

reclamation WWTP by the CAFLUX bioassay. They found levels of TEQ_{bio} in the range from 0.26 to 0.36 ng/l (24h CAFLUX exposure). These results are in good comparison to those observed in the present study (Table 41). In another study, Dagnino et al. (2010) detected much higher dioxin-like activity in effluents of 3 French municipal WWTPs (2.1–43.7 ng/l TEQ_{bio}). These values were based on relatively short and unusual 8h exposures, and the authors observed decrease of the activity during the prolonged exposure time. This is important to mention as our results were based on more commonly used 24h exposure. In another study Ma et al. (2005) did not find levels higher than 14 pg/l TEQ_{bio} in influents or effluents from Chinese municipal WWTPs using 72h exposure time. Taken together, it is apparent that WWTP effluents contain detectable amounts of AhR ligands but more research is needed to understand the role of dioxin-like compounds in dissolved phase of environmental mixtures in general.

Table 41: Comparison of the FATE SEES data for dioxin-like toxicity with literature data.

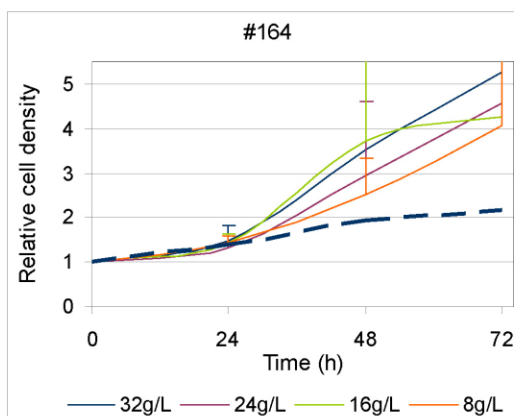
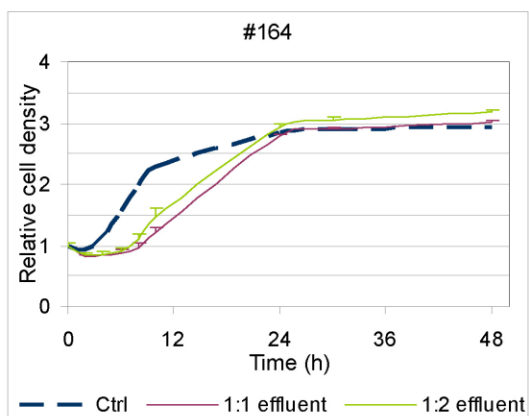
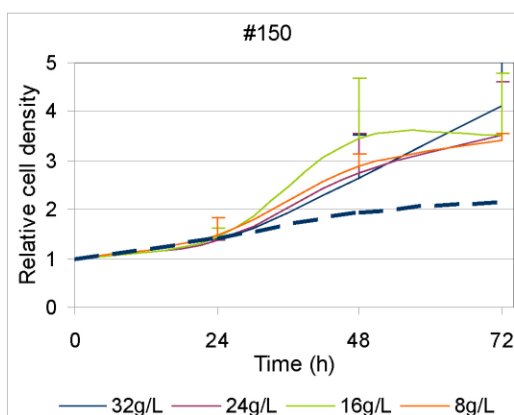
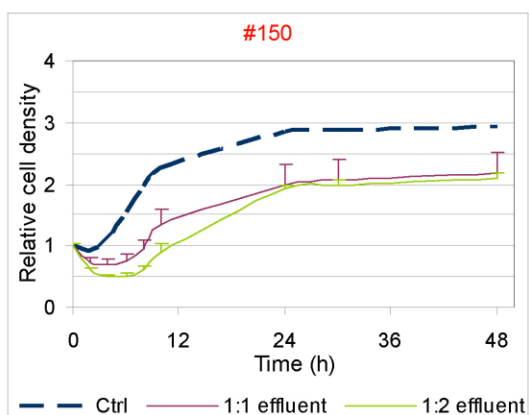
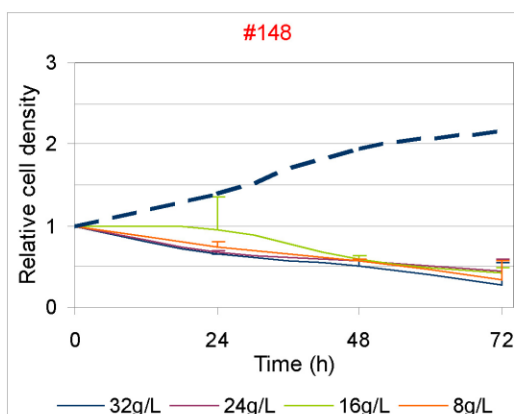
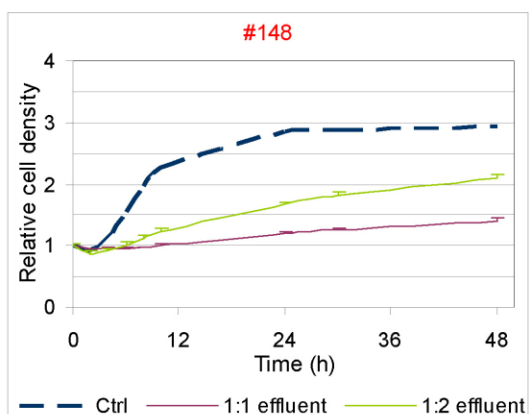
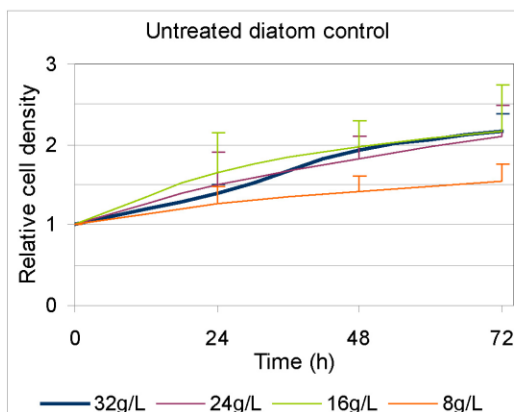
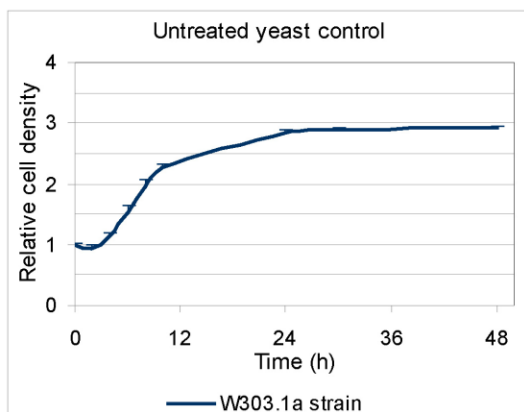
Location	Exposure time (h)	Median TEQ _{bio} (ng/l)	Average TEQ _{bio} (ng/l)	Max. TEQ _{bio} (ng/l)	LOD TEQ _{bio} (ng/l)	n WWTPs	Freq. (%)	Reference
EU wide study	24	0.14	0.14	0.44	0.10	25	84	FATE SEES
France	8	17.1	18.6	43.7	2 (LOQ)	3	100	Dagnino et al., 2010
Australia	24	0.31	n.a.	0.36	n.a.	1	100	Reungoat et al., 2010
China	72	<LOD	<LOD	<LOD	0.001	1	0	Ma et al., 2005

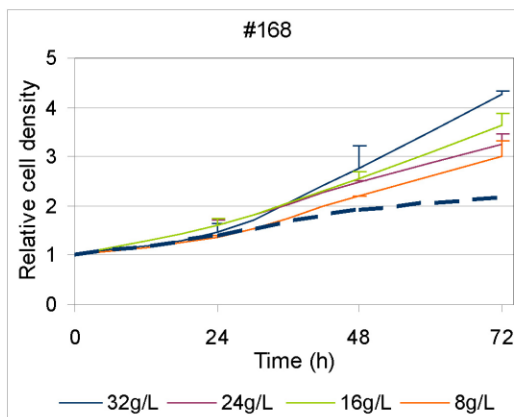
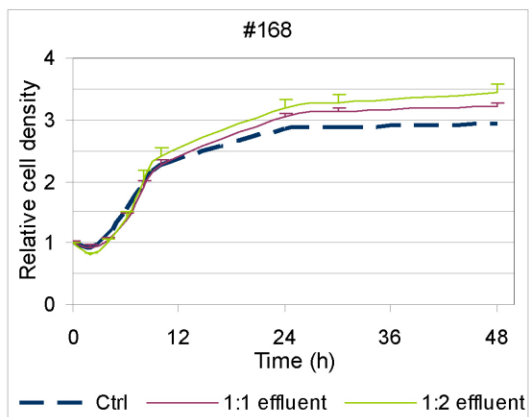
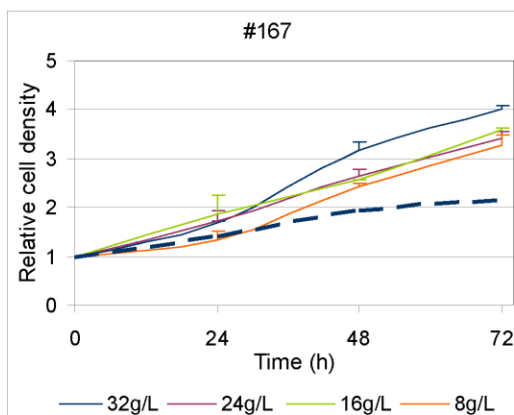
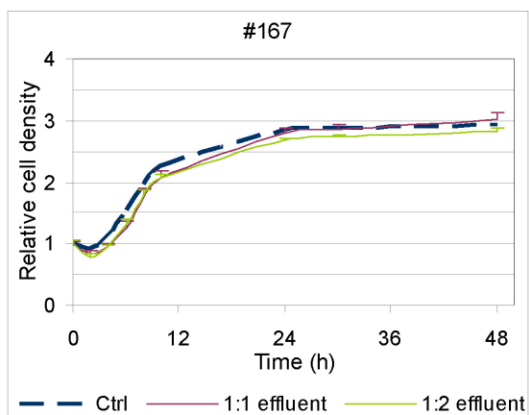
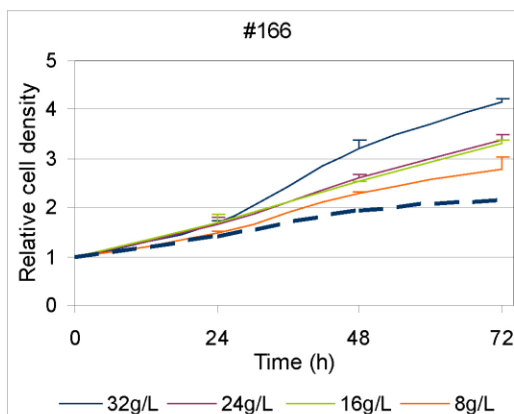
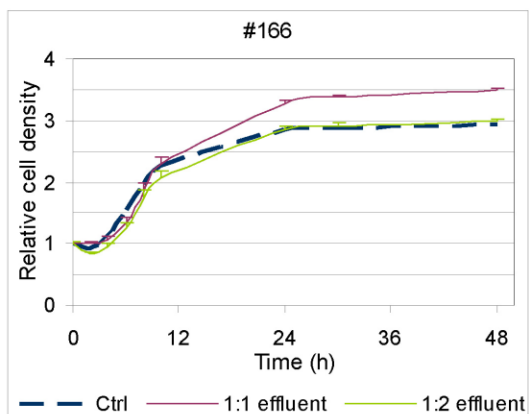
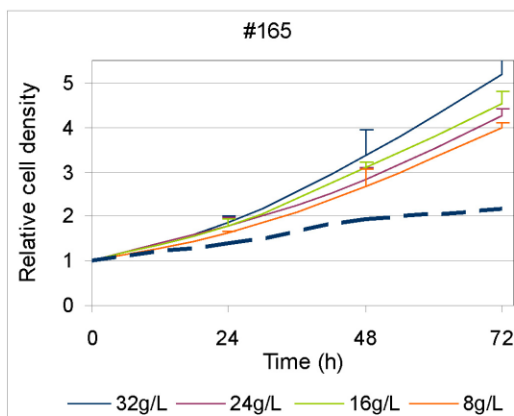
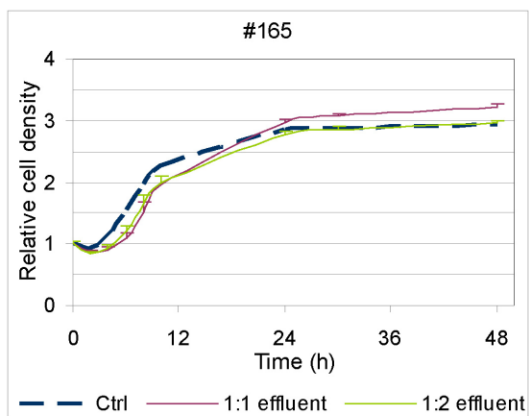
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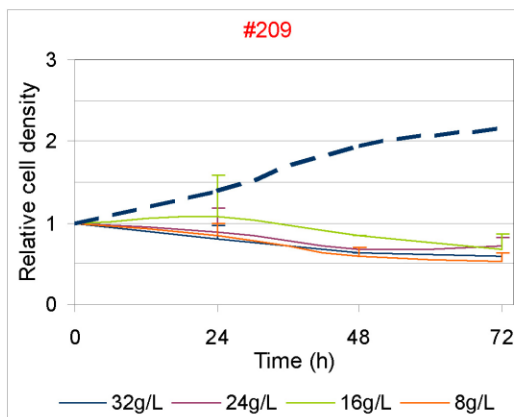
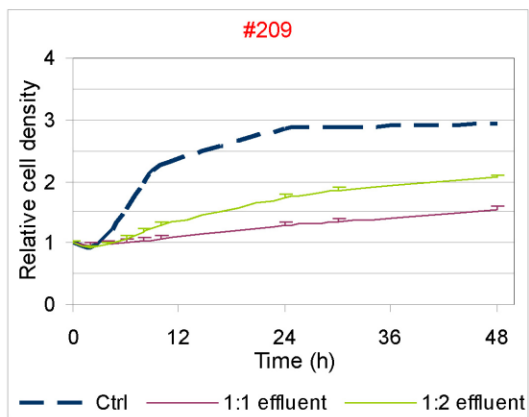
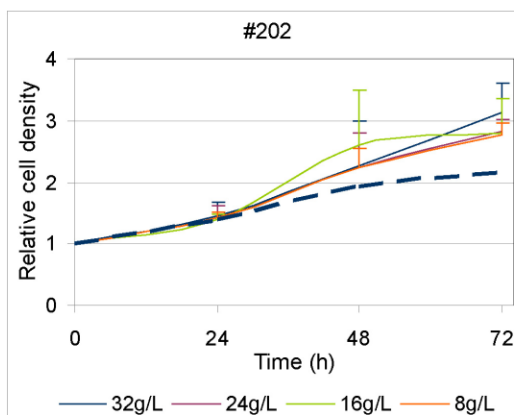
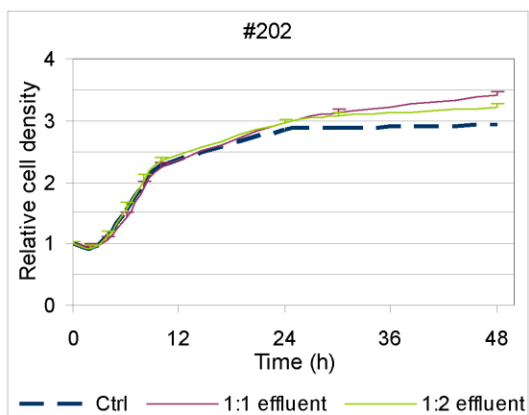
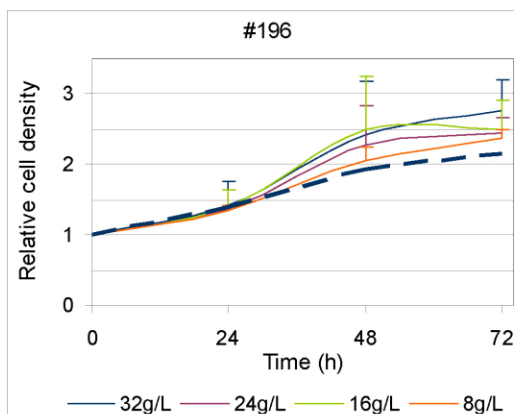
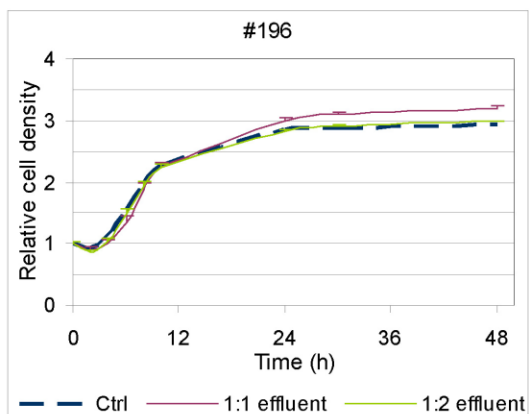
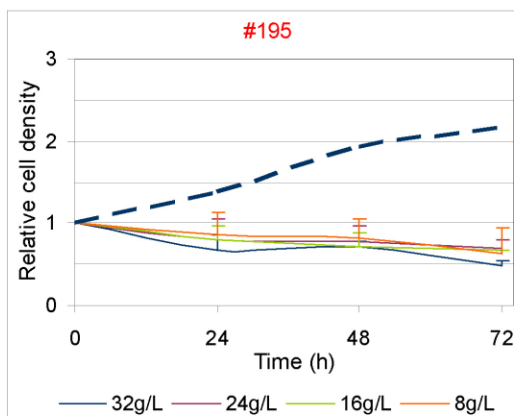
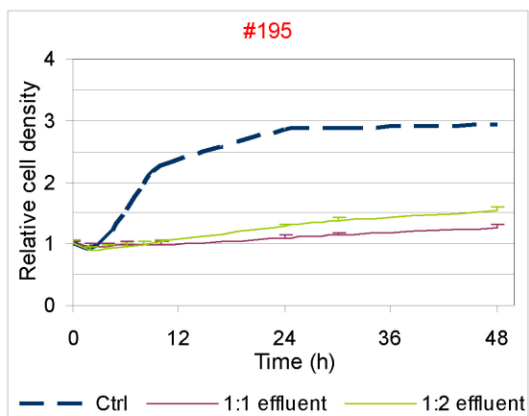
6.4.3. Acute toxicity to yeast and diatoms

The progressive adaptation of *Thalassiosira pseudonana* cultures to lower salinities was successfully achieved and the growth rates at 24 and 16 g/l Sodiumchloride (NaCl) after several weeks were comparable to those at 32 g/l, while a reduced growth rate was visible for 8 g/l, even after one month kept at lower salinity. The cultures at lower salinity had however corresponding higher sensitivity to additional stress and were able to identify toxicants at lower concentrations than the cultures at normal salinity. Additionally, the down-scale of both the yeast and diatom cultures to 96-well plate allowed fast screening of multiple samples simultaneously, greatly reducing the time and cost of the bioassay.

The effect elicited by the different effluents was quite comparable between yeast and diatom cultures. Most of the effluents were not harmful to any of the organisms. Some effluents even induced an increase in the growth of the cultures, more evidenced in the case of diatom, and for effluents # 165, 166, 167, 168 and 210 (Figure 8).







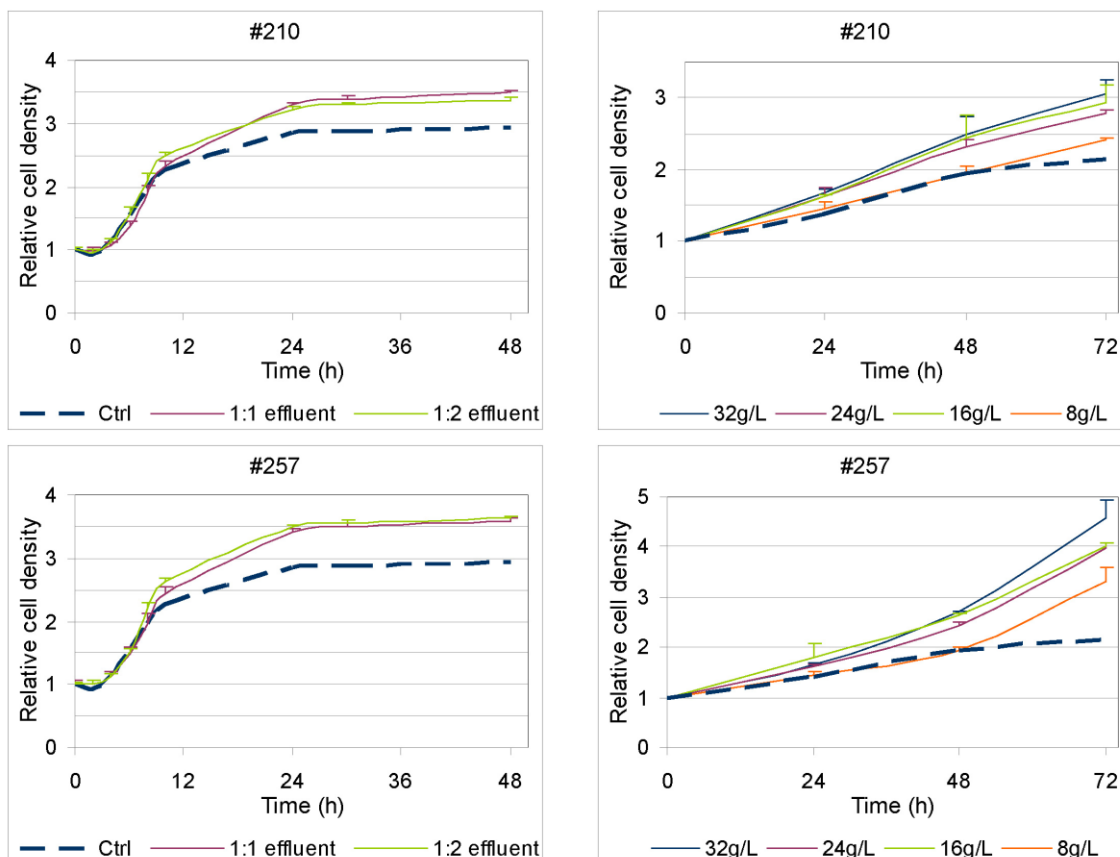


Figure 8: Effect of effluent exposure to culture growth of yeast *Saccharomyces cerevisiae* and diatom *Thalassiosira pseudonana*. The graphs show the relative cell density of the organisms monitored during exposure to 13 different effluent samples from waste water treatment plants. *S. cerevisiae*, W303.1a strain, was exposed to 1:1 (purple line) and 1:2 (green line) dilutions of effluent samples (left column), and *T. pseudonana*, growing at diverse salinities (32 g/l, blue; 24 g/l, purple; 16 g/l, green and 8 g/l, orange), were exposed to a 1:1 dilution of effluent samples (right column). The untreated yeast W303.1a strain cultures were used as control, while for diatom the untreated cultures grown at standard salinity (32 g/l; dashed blue line) was chosen as reference to all the treatments. Bars represent the standard deviation of the mean. In red the samples showing the growth inhibition effects.

The higher growth upon exposure to these effluents may indicate an abnormal enrichment in nutrients, which is particularly relevant to autotrophic organisms like diatom, and may be an indication of the eutrophication potential of the effluent water.

From the 13 effluent waters tested, only three revealed themselves harmful for the growth of both organisms, i.e. effluents #148, 195 and 209 (Figure 8, text in red). Both systems identified #195 as the most toxic effluent of those tested. While the effect of these effluents induced a growth inhibition of yeast cultures in a dose-response manner, they were lethal to diatom cultures of all salinities already after 24 hours exposure (Figure 8). When a dose-response was performed with these effluent samples on diatom cultures at different salinities, it was clear that sample #195 was the most toxic of all

samples. Thus, for the cultures at 32 g/l salinity, already at a 1:10 dilution, the #195 effluent induced a decrease in the growth rate by half, while at a dilution of 1:5 and lower it was lethal. For the effluents #148 and 209, the lethality was induced only at lower dilutions (1:3) of the effluent. The lower salinity cultures of this marine diatom, revealed themselves more sensitive to the effluents and the dose-response of the effluents performed at lower salinities confirmed the data obtained at normal salinity. At 24 g/l, effluents #148 and 209 were lethal for dilutions lower than 1:5 and almost completely eliminated the diatom growth for the dilution 1:10, while effluent #195 was lethal in all dilutions tested (Figure 9). For the lower salinities (16 and 8 g/l) the effluents were lethal in all dilutions tested (not shown).

Two additional effluents, while harmless to diatom, caused alterations in yeast growth, i.e. effluent #150 caused growth inhibition and #164 caused a delay in the lag-phase that rescued completely after 24h. In these cases, it is possible that the presence of nutrient may mask any effect that could be otherwise visible by pollutants in diatoms.

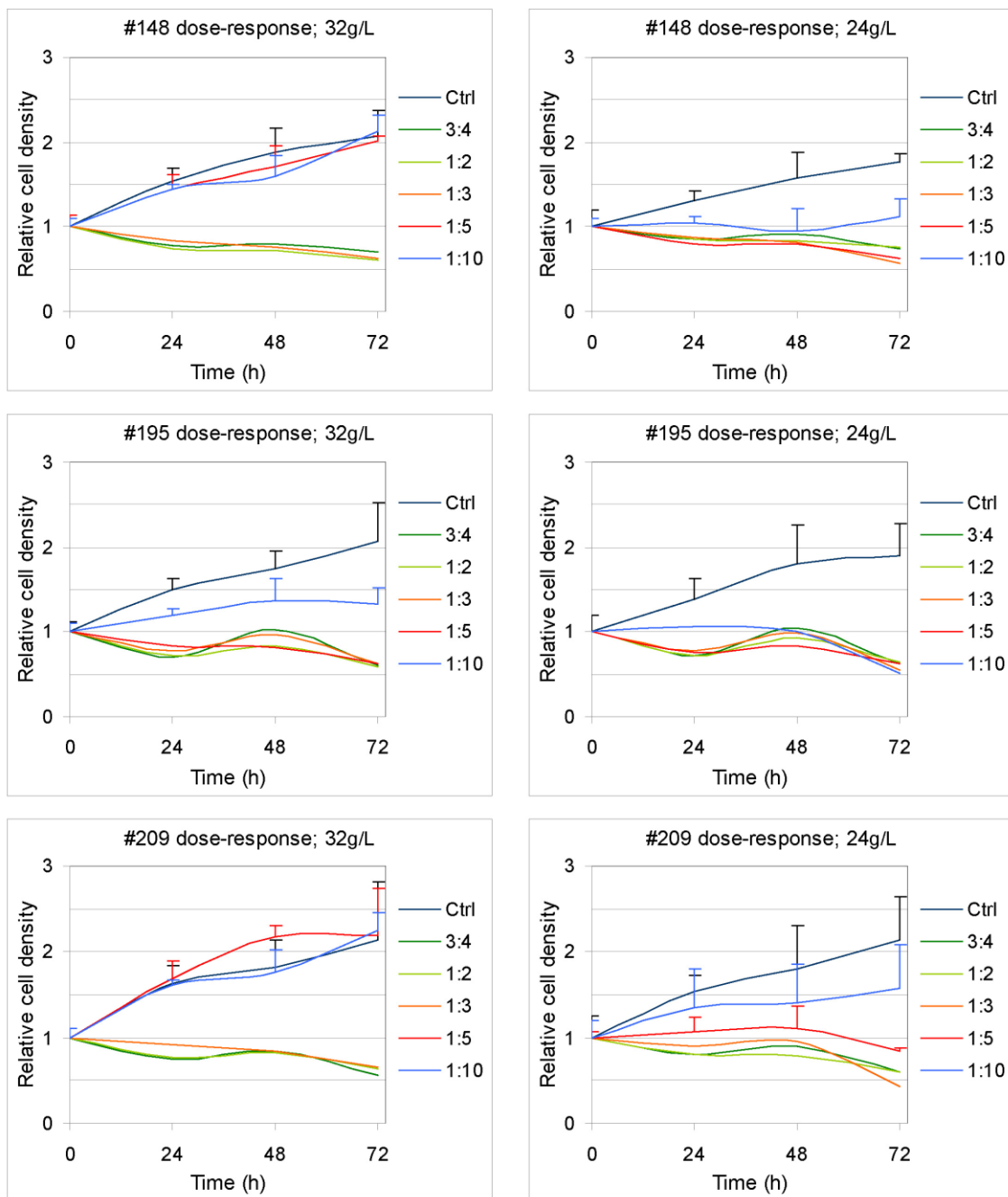


Figure 9: Determination of the lowest effluent dilution with harmful effect on the growth of *T. pseudonana*. Graphs show the relative cell density of diatom cultures growing at two different salinities, 32 g/l (left column) and 24 g/l (right column), upon exposure to the three toxic effluent samples. Five dilutions of the effluents were tested and compared to the untreated diatom cultures grown at the same salinities (Ctrl; dark blue line).

7. Conclusions and outlook

This European-wide monitoring study on the occurrence of micropollutants in waste water treatment plant (WWTP) effluents was the largest WWTP survey ever performed in Europe. The influent waters of the WWTPs were not analysed, therefore, no elimination rates for the chemicals were determined. What is clear, however, is that the elimination of most of the substances in conventional WWTPs with secondary biological treatment is incomplete and improvements of the wastewater treatment and subsequent treatments of the produced sludge are required to prevent the introduction of these micropollutants in the environment. It must be kept in mind that today's conventional waste water treatment technology (mechanical and biological steps) dates back to the 1970s, and that it was designed to remove nitrogen and phosphorus. Small polar organic molecules are poorly removed, many of the non-polar chemicals can however be removed with the sewage sludge.

Under the view of escalating population growth, and increased water stress in many regions of the world, reuse of treated water and waste water recycling are becoming more important options for water supply. It is being discussed in Europe to upgrade WWTPs with additional tertiary treatment steps such as ozonation and/or powdered activated carbon adsorption to remove micropollutants from WWTP effluents. In the Swiss "Micropoll Strategy" project complementary treatment steps have been evaluated and it has been shown that water quality can be significantly improved using processes such as powdered activated carbon adsorption or ozonation. A further treatment even to drinking water quality is possible by micro- and nanofiltration or reverse osmosis.

In the future, participation of even more laboratories and the analysis of additional chemicals in similar monitoring campaigns would be desirable. This report gives a good overview on important anthropogenic chemical substances relevant in WWTPs or surface waters. Large scale multi-compound analytical screening techniques are being developed, and LC-high-resolution-MS allows a general unknown screening in water samples. LC-MS libraries are necessary for the identification of unknown chemicals.

More detailed information on the single WWTPs would help in understanding also fate and behavior of the substances. Mass loads of the substances could be calculated if the effluent flows of the WWTPs were known. In addition, it is clear that better strategies are needed to integrate chemical and biological testing methods (such as biomarkers). Moreover, there is a need for more interlaboratory studies for the comparison of analytical method performances and QA/QC (e.g. for Diclofenac).

Biological-based tools, using acute and chronic toxicity testing of simple model organisms in the laboratory can provide fast and valuable information on the toxicity potential of the unprocessed water samples and infer possible effects onto endogenous species in the environment. In the report, we describe a fast, sensitive and easy method for fast screening of water samples based on acute toxicity using yeast or diatom. These bioassays however are not able to identify the specific chemical source of the effect. For

this purpose, posterior assessment of harmful water samples by chemical or molecular tools based on the mode of action of specific classes of pollutants could provide the link between the ecological effect and the class of compounds responsible for that effect.

The increasing worldwide contamination of freshwater systems with thousands of industrial and natural chemical compounds is one of the key environmental problems facing humanity. Although most of these compounds are present at low concentrations, many of them raise considerable toxicological concerns, particularly when present as components of complex mixtures ([Schwarzenbach et al., 2006](#)).

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9. Annex

Annex 1: European WWTP effluent water samples. Dom. = Domestic; Ind. = Industrial; Tert. = Tertiary Treatment.

No.	Sample Name	Country	Location	Sampling date	Extraction date (JRC)	Discharges	Plant Capacity [m3/d]	Capacity Population Equivalent	Tertiary Treatment Type
1	Fate Sees_136	Cyprus	Larnaka	12.05.2010	02.07.2010	Domestic (Dom.)	6000	27500	Tert. with sand filtration; chlorination
2	Fate Sees_137	Cyprus	Limassol	04.05.2010	01.07.2010	Dom.; Ind.	15000	70000	Tert. with sand filtration; chlorination
3	Fate Sees_138	France	Evieu (CA 6)		23.02.2011				
4	Fate Sees_139	France	Andancette (CA2)		23.02.2011				
5	Fate Sees_140	France			23.02.2011			13000	
6	Fate Sees_141	France			23.02.2011				
7	Fate Sees_142	France	Givors (CA5)		23.02.2011				
8	Fate Sees_143	Czech Republic	Not disclosed	10.05.2010	29.06.2010	Dom.; Ind.; no rain	700	5000	
9	Fate Sees_144	Czech Republic	Not disclosed	10.05.2010	28.06.2010	Dom.; Ind.; no rain	300	2500	
10	Fate Sees_145	Czech Republic	Not disclosed	09.05.2010	28.06.2010	Dom.; rain	3000	15000	
11	Fate Sees_146	Czech Republic	Not disclosed	09.05.2010	28.06.2010	Dom.; rain	19000	75000	
12	Fate Sees_147	Czech Republic	Not disclosed	11.05.2010	01.07.2010	Dom.; Ind.; rain	> 100000	>500000	
13	Fate Sees_148	Czech Republic	Not disclosed	09.05.2010	28.06.2010	Dom.; Ind.; rain	52000	170000	
14	Fate Sees_149	Czech Republic	Not disclosed	10.05.2010	28.06.2010	Dom.; Ind.; rain	> 200000	>500000	
15	Fate Sees_150	Belgium	Tankreining, Evergem (2406)	19.06.2010	15.07.2010	Industrial			
16	Fate Sees_151	Belgium	Shanks Vlaanderen, Lokeren	16.06.2010	14.07.2010	Industrial			
17	Fate Sees_152	Belgium	Truck-en Tankcleaning Tack, Oostrozebeke	23.06.2010	16.07.2010	Industrial			
18	Fate Sees_153	Belgium	Colortex, Sint-Niklaas	16.06.2010	16.07.2010	Industrial			
19	Fate Sees_155	Belgium	EOC Belgium, Oudenaarde	15.06.2010	15.07.2010	Industrial			
20	Fate Sees_156	Belgium	3M Belgium, Zwijndrecht	28.06.2010	14.07.2010	Industrial			
21	Fate Sees_157	Belgium	Taminco Gent (69471)	16.06.2010	14.07.2010	Industrial			
22	Fate Sees_158	Belgium	Ajijnomoto Omnicem, Wetteren	23.06.2010	16.07.2010	Industrial			
23	Fate Sees_159	Belgium	Bayer Antwerpen, Zandvliet	30.06.2010	16.07.2010	Industrial			
24	Fate Sees_160	Belgium	Janssen Pharmaceutica, Geel	16.06.2010	15.07.2010	Industrial			
25	Fate Sees_161	Belgium	Claerebout Potatoes, Heuvelland	17.06.2010	15.07.2010	Food industry			
26	Fate Sees_162	Belgium	Ardo, Ardooie (Expl. 84)	23.06.2010	15.07.2010	Food industry			
27	Fate Sees_163	Belgium	Agristo, Hulste (Expl. 369)	21.06.2010	09.07.2010	Food industry			
28	Fate Sees_164	Belgium	RWZI Ronse	17.06.2010	16.07.2010	Domestic			
29	Fate Sees_165	Belgium	RWZI Waregem	23.06.2010	14.07.2010	Domestic			
30	Fate Sees_166	Belgium	RWZI Deurne, Antwerpen	22.06.2010	14.07.2010	Domestic			
31	Fate Sees_167	Belgium	RWZI Hasselt, Kuringen	22.06.2010	15.07.2010	Domestic	11700	65000	
32	Fate Sees_168	Belgium	RWZI Geel	23.06.2010	14.07.2010	Domestic			
33	Fate Sees_169	Slovenia	Ljubljana	15.04.2010	01.07.2010	Dom. (62%); Ind. (11%); rain (21%)	103000	360000	
34	Fate Sees_170	Finland	Nummi-Pusula		05.07.2010				

35	Fate Sees_171	Finland	Lohja		05.07.2010				
36	Fate Sees_175	Hungary	Alattyán Municipal WWTP	08.07.2010	23.02.2011		250		
37	Fate Sees_177	Hungary	Martfű	08.07.2010	24.02.2011		1000		
38	Fate Sees_179	Finland	Vihti		05.07.2010				
39	Fate Sees_180	Finland	Mäntsälä		09.07.2010				
40	Fate Sees_181	Finland	Helsinki		06.07.2010				
41	Fate Sees_182	Finland	Espoo		08.07.2010				
42	Fate Sees_183	Greece	Thessaloniki (WWTP - EELTH)		06.07.2010				
43	Fate Sees_184	Greece	Thessaloniki (WWTP - EEL AINEIA)		07.07.2010				
44	Fate Sees_189	Switzerland	Wenslingen		09.07.2010				
45	Fate Sees_190	Switzerland	Seuzach	17.05.2010	08.07.2010		3940		
46	Fate Sees_191	Switzerland	Konolfingen	04.06.2010	08.07.2010				
47	Fate Sees_193	Switzerland	Affoltern a.A.		09.07.2010				
48	Fate Sees_194	Switzerland	Zürich Werdhölzli		01.07.2010				
49	Fate Sees_195	Ireland	Dublin	24.05.2010	01.07.2010		400000	1900000	Tert. UV Light Treatment
50	Fate Sees_196	Ireland	Osberstown	24.05.2010	02.07.2010				
51	Fate Sees_198	Austria	WV Hofsteig	08.06.2010	07.07.2010	Metal, food, landfill, textile, laundry	138150		
52	Fate Sees_199	Austria	AWV Region Feldkirch	08.06.2010	06.07.2010	Paper industry, food, metal, textile	380000		
53	Fate Sees_202	Austria	AWV Hall i.Tirol-Fritzens	08.06.2010	08.07.2010		16000	120000	
54	Fate Sees_203	Austria	Eisenstadt	18.05.2010	29.06.2010			42000	
55	Fate Sees_204	Austria	AWV Wiener Neustadt - Sud	24.05.2010	28.06.2010	Domestic (90%); Paper Ind.		260000	
56	Fate Sees_205	Austria	Feldkirchen	19.05.2010	29.06.2010		6640	50000	
57	Fate Sees_208	Spain	Tortosa	14.04.2010	29.06.2010		10296	46847	
58	Fate Sees_209	Spain	Ulldecona	14.04.2010	29.06.2010		1620	13500	
59	Fate Sees_210	Spain	Godall	14.04.2010	29.06.2010		150	875	
60	Fate Sees_211	Sweden	Ryaverket		24.02.2011	Dom.;Ind.; hospital; laundries; food ind.	119000000	680000	
61	Fate Sees_214	Sweden	Henriksdal		24.02.2011	Dom.;Ind.; hospital; laundries; food ind.	88000000	900000	
62	Fate Sees_226	Italy	WWTP Roma nord ACEA	25.05.2010	02.07.2010		354240	780000	Final disinfection step
63	Fate Sees_227	Lithuania	WWTP Klaipėda vanduo	19.05.2010	05.07.2010	Domestic			
64	Fate Sees_228	Lithuania	Kaunas	18.05.2010	02.07.2010		82000		
65	Fate Sees_229	Lithuania	Panevezys regional	19.05.2010	01.07.2010		70000		
66	Fate Sees_233	NL	WWTP Harnaschpolder	09.06.2010	06.07.2010		150000	1400000	
67	Fate Sees_235	NL	WWTP Zaandam Oost	03.06.2010		Dom.; urban runoff; Ind.; craft industry			
68	Fate Sees_236	NL	Rotterdam Dokhaven	19.05.2010	02.07.2010	Mainly Domestic	117808	500000	
69	Fate Sees_239	NL	WWTP Venlo	22.06.2010	07.07.2010				
70	Fate Sees_242	NL	WWTP Winterswijk		05.07.2010	Dom.; Ind. (30-40%); hospital		83500	
71	Fate Sees_243	NL	WWTP Nieuwgraaf		07.07.2010	Dom.; Ind. (30-40%); hospital		395000	
72	Fate Sees_245	NL	WWTP Simpelveld		24.02.2011	Dom.; health care unit		20500	
73	Fate Sees_247	NL	WWTP Amstelveen	14.06.2010	07.07.2010	Domestic		125000	
74	Fate Sees_248	NL	Almere	18.05.2010	02.07.2010	Domestic; hospital; no rain		330000	
75	Fate Sees_250	Germany	Klaranlage Seehausen, Bremen	17.06.2010	05.07.2010		94246	1000000	

76	Fate Sees_252	Germany	Klärwerk Gut Marienhof (Muenchen)						
77	Fate Sees_253	Italy	Depuratore 'Jugendwerk Brebbia'		07.07.2010				
78	Fate Sees_254	Portugal	WWTP Parada		24.02.2011				
79	Fate Sees_256	Germany	AZV Hungerbachtal	28.06.2010	09.07.2010				
80	Fate Sees_257	NL	WWTP Leek (Noorderzijlvest)		06.07.2010	Domestic		34000	
81	Fate Sees_258	NL	WWTP Garmerwold (Noorderzijlvest)		06.07.2010	Domestic		300000	
82	Fate Sees_259	Portugal	WWTP Viana do Castelo	25.06.2010	08.07.2010				
83	Fate Sees_260	Sweden	Umeå		24.02.2011	Dom.; Ind.; hospital; dairy	35342	116000	
84	Fate Sees_261	Sweden	Nolhaga		24.02.2011	Dom.; Ind.; laundry	8767	60000	
85	Fate Sees_262	Sweden	Gasslosa		24.02.2011	Dom.; Ind.; hospital; food;; laundry; craft ind.	3452	110000	
86	Fate Sees_263	Sweden	Ellinge		24.02.2011	Dom.; Ind.; food; crop; laundry; craft ind.	10137	330000	
87	Fate Sees_264	Sweden	Borlange		24.02.2011	Dom.; Ind.; hospital; paper mill	15342	60000	
88	Fate Sees_265	Sweden	Bollebygd		24.02.2011	Dom.; Ind.; fluggers process water	658	6000	
89	Fate Sees_266	Sweden	Bergkvara		25.02.2011	Dom.	1644	6500	
90	Fate Sees_267	Sweden	Framby		25.02.2011	Dom.; Ind.; hospital; laundry; craft ind.	18904	50000	
91	Fate Sees_268	Sweden			25.02.2011	Dom.; Ind. Ironworks; paper mill; hospital, laundry, slaughterhouse; craft ind.	7123	30000	

Annex 2: Analytical results for the European WWTP effluent water samples in [ng/l]. Lab: JRC-IES. “Higher” concentrations are in red; clean, unpolluted water samples are with yellow background; “Zero” means below LOQ.

Sample	Sucralose [µg/l]	Acesulfame [µg/l]	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFBS	PFHxS	PFOS	Diclofenac*	MCPA	Mecoprop	2,4-D	Ketoprofen	Naproxen	Ibuprofen
LOQ [ng/l]	0.1 µg/l	0.01 µg/l	1	1	1	0.5	1	1	1	0.5	1	0.5	1	1	10	5	2
Fate Sees_136	1,33	1,89	0,0	4,5	11,8	3,8	3,0	0,0	0,0	6,2	21,6	0,0	119,4	8,0	56,7	18,0	163,0
Fate Sees_137	6,07	15,63	13,9	3,9	12,1	1,8	2,4	0,0	4,5	4,7	57,0	0,0	0,0	13,7	36,0	267,4	117,9
Fate Sees_138	2,05	96,21	0,0	9,3	24,3	4,9	3,4	6	2,4	30,6	3,5	3,2	0,0	1,8	171,4	0,0	0,0
Fate Sees_139	0,93	44,81	8,1	3,9	5,3	1,2	1,1	0,0	2,6	7,5	12,2	0,0	0,0	0,0	65,5	46,9	0,0
Fate Sees_140	2,65	68,04	31,8	17,4	31,5	9,3	7,6	0,0	8,8	22,3	69,5	6,9	31,7	44,5	190,3	5,2	0,0
Fate Sees_141	1,68	54,88	8,5	8,4	17,0	6,4	5,3	0,0	4,7	28,9	50,0	1,8	17,6	28,0	724,7	68,8	2128,5
Fate Sees_142	4,17	184,7	92,2	37,6	60,8	7,9	8,4	28	180,2	527,7	48,7	28,5	111,4	41,7	63,3	17,2	45,1
Fate Sees_143	1,60	18,24	7,4	5,1	10,7	1,8	2,5	60	0,0	8,9	85,7	35,7	15,8	43,3	21,6	22,4	89,5
Fate Sees_144	4,52	88,22	7,6	3,6	14,8	0,0	0,0	65	0,0	0,7	80,4	394,5	0,0	21,4	30,7	5,4	0,0
Fate Sees_145	2,34	94,77	8,3	4,4	12,6	1,9	0,6	127	0,0	1,4	88,1	217,7	14,4	27,4	63,2	14,8	0,0
Fate Sees_146	1,46	73,74	14,8	6,1	10,7	0,7	2,0	37	0,0	18,1	89,4	30,9	28,5	37,7	104,0	9,8	7,2
Fate Sees_147	1,42	14,31	8,4	4,3	35,0	2,4	10,6	26	0,0	12,4	67,9	50,1	25,4	29,7	118,9	24,8	0,0
Fate Sees_148	1,71	39,48	14,9	11,0	12,2	2,1	0,0	84	16,5	25,3	49,8	55,8	2208,6	22,4	1653,1	0,0	43,6
Fate Sees_149	2,77	61,17	6,2	8,5	8,7	1,7	3,1	25	2,7	25,2	74,9	25,5	182,0	30,5	32,5	24,7	124,7
Fate Sees_150	3,78	0,00	1257,0	1926,3	1185,2	99,0	4,3	7476	921,9	113,9	84,0	256,5	1126,5	356,7	0,0	0,0	164,8
Fate Sees_151	0,00	0,41	1102,9	293,8	153,9	3,6	1,9	5087	459,4	78,3	0,0	14,7	65,5	11,8	0,0	0,0	0,0
Fate Sees_152	0,68	1,71	180,7	219,6	1307,1	11,0	5,9	9120	332,0	77,1	23,0	33,3	106,8	25,0	0,0	0,0	0,0
Fate Sees_153	0,00	0,00	0,0	1,1	11,1	0,0	0,0	0,0	0,0	0,0	4,8	0,0	0,0	106,5	0,0	0,0	18,1
Fate Sees_155	0,00	0,30	0,0	0,0	119,9	0,0	0,0	0,0	0,0	0,0	0,0	41,5	0,0	0,0	0,0	0,0	5,6
Fate Sees_156	0,00	0,16	23866,5	2962,3	226,9	0,8	0,0	3200000	868,5	139,9	0,0	0,0	0,0	0,0	0,0	0,0	4,4
Fate Sees_157	0,00	0,00	0,0	0,0	13,0	5,6	2,7	0,0	15,9	21,4	1,9	181,1	101,6	74,4	0,0	0,0	7,9
Fate Sees_158	0,00	0,11	0,0	22,7	12,3	5,2	0,0	0,0	13,3	0,0	0,9	53,8	0,0	0,0	0,0	0,0	29,3
Fate Sees_159	0,00	0,00	0,0	6,3	10,9	0,0	0,0	23	0,0	33,4	0,0	0,0	635,7	0,0	0,0	22,3	25,1
Fate Sees_160	0,00	4,56	16,1	4,4	12,8	0,9	2,3	41	206,7	147,8	0,0	0,0	0,0	136,4	0,0	0,0	0,0
Fate Sees_161	0,00	0,00	0,0	4,6	3,1	0,0	4,1	0,0	0,0	3,3	0,0	7,7	4,6	0,0	425,3	0,0	0,0
Fate Sees_162	0,00	0,37	4,0	0,0	3,9	1,2	0,0	56	0,0	8,3	0,0	0,0	0,0	3,9	0,0	0,0	0,0
Fate Sees_163	0,00	0,00	0,0	2,2	3,8	8,5	0,0	0,0	1,2	2,6	3,3	16,7	0,0	0,0	154,1	0,0	0,0
Fate Sees_164	1,48	0,88	0,0	40,8	242,2	63,6	132,3	0,0	0,0	91,9	82,0	462,7	211,7	80,2	0,0	69,9	225,6
Fate Sees_165	2,18	13,69	116,0	1079,6	2534,0	2734,5	1687,0	34	0,0	11,9	33,8	90,1	180,7	42,9	0,0	9,1	0,0
Fate Sees_166	2,81	10,03	231,4	425,0	15939,7	22,4	39,4	24	30,9	39,4	56,9	18,5	190,3	24,2	122,6	6,3	0,0
Fate Sees_167	3,27	17,93	3,3	2,8	12,3	2,2	3,6	27	4,2	4,3	79,6	749,7	79,5	58,0	0,0	17,9	0,0
Fate Sees_168	1,44	10,96	0,0	8,1	27,6	2,8	0,0	2529	137,6	2100,9	39,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_169	1,83	6,39	5,4	4,0	13,3	0,7	3,3	33	3,0	28,4	68,3	0,0	74,3	11,4	0,0	2,6	0,0
Fate Sees_170	4,98	11,07	0,0	4,0	4,9	3,4	0,0	0,0	0,0	2,4	133,8	12,7	16,7	0,0	443,2	957,6	70,5
Fate Sees_171	1,56	9,02	0,0	5,1	7,3	0,8	0,0	28	31,6	9,9	43,0	0,0	11,8	0,0	94,3	54,7	21,4
Fate Sees_175	0,20	2299	11,1	0,0	1,7	1,4	2,7	0,0	3,6	1,8	2,0	1,0	0,0	27,5	168,4	7,2	19,7
Fate Sees_177	0,44	2482	8,9	3,0	5,4	0,0	0,0	0,0	2,4	4,4	10,8	0,7	7,0	19,3	64,3	0,0	78,3
Fate Sees_179	6,24	33,26	4,9	6,4	15,9	5,4	5,5	41	0,0	0,0	78,2	6,9	8,6	0,0	0,0	0,0	4,6
Fate Sees_180	3,06	29,33	4,8	6,1	19,1	2,8	1,8	70	19,7	15,9	35,7	218,3	120,7	0,0	0,0	5,5	0,0
Fate Sees_181	4,99	32,69	5,4	6,4	14,7	4,3	4,8	0,0	10,9	240,6	46,3	25,7	44,5	9,4	149,1	28,7	16,4
Fate Sees_182	4,50	22,01	9,2	8,3	21,5	2,1	2,6	43	7,2	12,4	44,7	25,3	103,4	10,1	138,4	15,7	0,0
Fate Sees_183	2,53	9,28	0,0	4,5	14,7	2,8	4,2	0,0	0,0	10,8	107,1	0,0	0,0	4,9	0,0	15,4	0,0
Fate Sees_184	2,92	6,54	5,7	3,1	12,1	0,0	2,0	0,0	2,7	7,2	0,0	0,0	0,0	0,0	0,0	0,0	8,5
Fate Sees_189	1,72	12,78	3,9	1,8	7,9	8,0	3,6	20	6,3	13,5	66,7	546,5	78,4	6,9	0,0	16,9	0,0
Fate Sees_190	2,42	11,71	3,6	5,1	24,5	3,1	8,1	47	0,0	19,6	81,1	12,2	0,0	20,1	0,0	9,1	0,0
Fate Sees_191	1,66	11,37	0,0	2,3	8,0	3,8	4,5	29	5,3	11,4	59,4	11,2	130,5	0,0	0,0	26,0	0,0
Fate Sees_193	1,74	10,40	0,0	4,8	15,2	1,9	4,5	48	8,9	11,4	68,2	16,6	78,0	9,2	0,0	20,7	0,0

Fate Sees_194	3,07	13,11	7,1	6,4	12,9	2,0	8,9	19	7,8	0,0	92,1	5,8	126,9	15,8	72,8	40,0	18,3
Fate Sees_195	7,13	12,41	4,3	6,4	18,5	2,3	5,5	0,0	3,7	6,6	80,3	58,8	193,4	62,0	0,0	30,1	77,2
Fate Sees_196	9,70	21,02	7,9	4,3	14,8	2,4	2,7	62	9,1	11,6	144,3	81,9	250,6	30,6	90,7	0,0	208,7
Fate Sees_198	1,09	5,80	11,2	11,2	24,6	3,8	0,0	76	14,2	32,2	106,2	35,8	229,6	27,4	0,0	0,0	8,4
Fate Sees_199	0,75	4,87	6,2	7,3	17,2	4,0	5,0	96	16,2	154,2	69,0	33,8	126,9	57,9	0,0	0,0	28,9
Fate Sees_202	1,96	8,47	3,7	2,7	7,8	2,5	3,3	2371	0,0	45,0	50,1	71,2	16,1	6,1	0,0	0,0	0,0
Fate Sees_203	0,49	7,22	4,6	5,3	0,0	3,6	3,2	34	7,5	9,7	76,7	536,3	129,9	31,9	73,5	25,5	16,3
Fate Sees_204	0,42	22,84	5,2	9,7	18,4	3,8	6,9	709	2,5	11,3	89,6	643,3	215,5	29,3	69,9	32,3	183,8
Fate Sees_205	2,28	14,33	7,1	5,4	10,2	2,5	2,6	41	8,1	155,2	174,3	5,5	7,4	0,0	178,5	0,0	0,0
Fate Sees_208	4,85	16,56	8,6	6,0	16,6	1,7	2,3	55	0,0	4,6	46,9	735,5	34,4	102,5	29,3	8,8	0,0
Fate Sees_209	3,74	12,40	6,8	3,7	7,5	2,3	3,1	11	0,0	1,8	29,9	1179,5	6,3	21,0	0,0	20,5	94,7
Fate Sees_210	2,08	4,04	0,0	4,3	7,0	1,9	0,0	14	0,0	2,3	42,3	2403,9	0,0	71,9	112,2	29,2	0,0
Fate Sees_211	0,94	43,35	2,7	5,8	9,7	1,1	1,1	0,0	3,0	14,4	14,4	0,5	0,0	0,0	191,1	19,6	4,4
Fate Sees_214	1,42	43,77	7,6	7,9	12,8	1,9	4,0	6	4,9	26,3	11,3	0,0	4,4	2,2	34,6	3,2	0,0
Fate Sees_226	4,35	17,62	8,9	8,5	17,7	5,0	1,6	0,0	18,1	16,0	1,7	0,0	0,0	0,0	0,0	55,6	0,0
Fate Sees_227	0,47	0,75	0,0	3,8	3,6	0,7	0,0	14	0,0	0,0	34,6	17,4	0,0	0,0	0,0	0,0	0,0
Fate Sees_228	1,06	6,14	5,7	3,0	10,1	2,0	4,1	59	0,0	4,1	166,3	394,8	10,4	109,8	83,6	10,7	131,0
Fate Sees_229	0,56	3,03	0,0	3,4	4,7	1,3	2,2	33	0,0	4,3	106,4	494,8	13,1	26,7	0,0	6,7	87,1
Fate Sees_233	6,57	35,31	7,8	7,6	29,7	4,0	6,4	14	34,6	32,2	34,1	323,0	106,8	86,2	0,0	7,8	35,6
Fate Sees_235	5,47	13,45	24,9	12,7	33,6	3,6	4,2	132	770,1	738,0	23,3	157,5	209,4	14,3	0,0	0,0	0,0
Fate Sees_236	7,09	31,12	10,7	13,8	39,7	3,9	1,8	18	36,3	42,0	50,5	31,4	89,4	2,3	0,0	27,7	29,4
Fate Sees_239	3,11	18,01	4,9	4,2	12,4	2,9	4,1	21	21,1	54,7	30,1	310,3	530,2	80,7	0,0	6,8	3,5
Fate Sees_242	5,13	13,73	0,0	5,2	15,6	3,4	3,4	0,0	3,2	8,1	37,2	96,5	513,4	0,0	0,0	12,5	16,6
Fate Sees_243	6,13	34,37	11,6	8,1	26,1	2,4	5,7	0,0	10,5	26,3	41,0	783,8	278,0	70,8	0,0	7,8	0,0
Fate Sees_245	2,10	100,3	11,4	2,7	11,1	0,0	2,2	0,0	1,0	4,5	15,4	61,1	172,9	12,1	0,0	2,8	0,0
Fate Sees_247	9,84	45,29	17,4	8,1	26,9	3,9	7,4	62	21,0	28,5	59,4	100,5	100,6	0,0	0,0	18,9	25,5
Fate Sees_248	8,55	25,83	13,5	10,6	61,7	4,9	15,7	47	8,2	9,8	38,3	103,2	188,8	50,3	0,0	8,6	22,6
Fate Sees_250	1,59	6,25	5,7	0,0	12,9	2,2	4,2	28	6,6	13,5	138,0	125,9	261,2	18,2	0,0	14,8	77,2
Fate Sees_252		0,00															
Fate Sees_253	12,88	4,89	0,0	2,1	2,0	0,0	1,7	0,0	0,0	1,7	59,6	0,0	0,0	0,0	0,0	27,2	205,0
Fate Sees_254	3,88	40,55	0,0	3,7	7,0	1,4	2,9	0,0	1,2	13,1	82,9	19,0	8,5	12,3	93,6	15,6	1889,5
Fate Sees_256	1,69	19,27	7,8	3,8	22,3	3,3	1,9	65	2,2	5,4	133,5	74,4	6,0	0,0	0,0	0,0	53,0
Fate Sees_257	5,26	13,33	0,0	3,4	9,5	2,5	3,2	0,0	1,6	3,6	33,2	648,8	1057,5	70,8	0,0	4,5	0,0
Fate Sees_258	6,10	22,87	9,3	6,1	30,1	6,0	18,6	9	7,4	26,0	24,6	265,9	354,0	0,0	0,0	0,0	0,0
Fate Sees_259	5,56	6,73	0,0	4,6	3,6	0,9	0,0	0,0	0,0	1,4	102,5	9,0	0,0	6,8	385,9	55,4	29,9
Fate Sees_260	1,11	41,50	6,1	7,9	10,2	1,2	5,4	0,0	2,5	8,6	0,0	1,1	0,0	13,6	130,8	29,8	75,1
Fate Sees_261	0,53	28,04	3,2	6,9	11,7	1,1	3,9	0,0	1,3	5,3	19,4	1,1	47,5	1,3	239,7	63,2	311,1
Fate Sees_262	0,50	21,77	14,4	20,1	35,9	8,1	7,2	3	10,1	44,5	13,1	3,9	10,2	7,1	77,2	5,3	0,0
Fate Sees_263	0,33	24,55	4,5	4,6	8,3	0,0	2,6	0,0	2,0	4,2	7,8	14,1	3,8	4,5	133,1	0,0	6,9
Fate Sees_264	0,61	36,60	4,2	4,8	7,5	1,4	1,0	0,0	3,2	15,3	18,7	1,5	10,4	2,5	221,9	17,5	0,0
Fate Sees_265	1,35	51,19	0,0	2,4	7,3	1,3	2,0	0,0	0,9	3,0	40,6	1,3	0,0	1,0	147,1	27,9	41,3
Fate Sees_266	0,87	61,25	0,0	5,7	5,7	1,1	1,2	0,0	10,1	14,9	20,6	1,9	0,0	0,0	52,2	0,0	0,0
Fate Sees_267	0,65	27,19	6,6	3,6	13,8	1,9	2,3	0,0	4,1	19,9	0,0	0,0	7,7	0,0	71,8	13,3	93,0
Fate Sees_268	0,91	38,29	7,8	9,7	17,2	2,0	6,0	3	4,2	18,1	43,7	0,0	12,3	2,1	156,3	0,0	0,0

*Diclofenac: It might be possible that the levels for Diclofenac are higher than reported here (see section 6.3.3.)

Annex 3: Results; Lab: JRC-IES, and IWW, Germany (X-ray contrast media and Gadolinium-compounds). "0.0" means below LOQ.

Sample	Bezafibrate	Gemfibrozil	Bentazone	Triclosan	Dichlorprop	Nitrophenol	Clofibric acid	Dinitrophenol	2,4,5-T	Amidotrizoic acid [µg/L]	Iohexol [µg/L]	Iomeprol [µg/L]	Iopamidol [µg/L]	Iopromid [µg/L]	Gadolinium [ng/L]	Gd-Anomaly	Anthropogenic Gd [%]
LOQ [ng/l]	1	1	1	4	5	5	2	5	2	0.05 µg/L	0.05 µg/L	0.05 µg/L	0.05 µg/L	0.05 µg/L	1.9	1.05	5
Fate Sees_136	9,6	112,1	0,0	46,5	0,0	104,2	0,0	50,0	0,0	0,55	0,75	< 0,05	< 0,05	0,19	58,35	33,5	97%
Fate Sees_137	44,6	272,3	0,0	0,0	0,0	57,9	0,0	0,0	0,0	1,6	0,28	< 0,05	< 0,05	1,2	41,05	51,4	98%
Fate Sees_138	0,0	3,1	0,0	0,0	0,0	27,7	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_139	0,9	0,0	0,0	33,4	0,0	41,4	0,0	10,8	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_140	5,6	0,0	0,0	0,0	0,0	63,9	0,0	23,9	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_141	343,5	17,9	3,7	176,8	4,0	114,9	0,0	25,7	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_142	124,6	12,8	0,0	163,5	0,0	167,2	127,0	13,3	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_143	0,0	0,0	5,4	0,0	8,9	31,8	16,3	68,9	0,0	< 0,05	< 0,05	0,1	< 0,05	< 0,05	2,30	1,5	32%
Fate Sees_144	38,4	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	43,90	52,5	98%
Fate Sees_145	0,0	0,0	1,4	97,5	0,0	14,5	0,0	59,9	0,0	< 0,05	< 0,05	< 0,05	< 0,05	0,72	1,80	2,0	51%
Fate Sees_146	0,0	1,2	12,2	0,0	0,0	0,0	0,0	38,6	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	5,65	9,4	89%
Fate Sees_147	1,5	1,1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	0,9	< 0,05	0,35	82,15	3,5	71%
Fate Sees_148	0,0	0,0	220,0	16,7	186,3	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	56,35	20,6	95%
Fate Sees_149	2,8	17,3	0,0	84,9	0,0	82,4	0,0	79,6	0,0	< 0,05	0,44	9,6	< 0,05	5,8	115,00	70,5	99%
Fate Sees_150	196,9	0,0	104,0	0,0	107,2	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	102,90	4,9	80%
Fate Sees_151	0,0	0,0	28,9	0,0	9,5	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	83,55	3,9	74%
Fate Sees_152	0,0	0,0	22,3	18,5	97,9	0,0	8,9	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	125,10	1,5	34%
Fate Sees_153	0,0	0,0	0,0	4258,6	0,0	599,6	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	46,05	1,0	< 5%
Fate Sees_155	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	54,15	1,5	33%
Fate Sees_156	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	58,50	8,1	88%
Fate Sees_157	3,1	4,9	20,4	0,0	57,3	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	35,00	1,3	24%
Fate Sees_158	0,0	4,1	0,0	0,0	0,0	20,1	4,6	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	17,35	18,5	95%
Fate Sees_159	0,0	0,0	0,0	0,0	0,0	132,8	0,0	473,5	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	34,45	1,9	46%
Fate Sees_160	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	16,90	2,4	58%
Fate Sees_161	0,0	0,0	7,2	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	15,70	185,8	99%
Fate Sees_162	0,0	0,0	22,0	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	15,90	4,7	79%
Fate Sees_163	0,0	0,0	2,5	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	17,45	21,5	95%
Fate Sees_164	19,8	0,0	17,0	23,9	0,0	0,0	0,0	0,0	0,0	5,2	< 0,05	< 0,05	< 0,05	< 0,05	218,75	23,6	96%
Fate Sees_165	0,0	0,0	12,0	0,0	15,8	0,0	0,0	0,0	0,0	1,7	< 0,05	< 0,05	< 0,05	< 0,05	154,05	95,5	99%
Fate Sees_166	2,6	2,1	0,0	26,2	0,0	0,0	11,6	0,0	0,0	0,31	< 0,05	0,11	< 0,05	0,44	418,90	87,9	99%
Fate Sees_167	0,0	5,0	2,5	16,7	18,1	0,0	3,0	0,0	0,0	3,7	< 0,05	< 0,05	< 0,05	0,09	389,45	18,0	94%
Fate Sees_168	2,2	0,0	3,4	0,0	0,0	576,3	0,0	0,0	0,0	0,64	< 0,05	< 0,05	< 0,05	< 0,05	154,75	9,0	89%
Fate Sees_169	0,0	102,2	16,9	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	0,25	1,1	0,65	1,3	49,60	60,5	98%
Fate Sees_170	6,8	12,0	0,0	0,0	37,2	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	710,50	107,5	99%
Fate Sees_171	9,9	0,0	0,0	0,0	0,0	14,1	0,0	0,0	0,0	0,5	< 0,05	0,25	< 0,05	< 0,05	23,55	7,6	87%
Fate Sees_175	0,0	0,0	0,0	10,4	0,0	68,6	0,0	0,0	0,0	5,8	< 0,05	0,84	< 0,05	< 0,05	65,10	1,0	< 5%
Fate Sees_177	0,0	0,0	0,0	4,2	0,0	1300,8	0,0	227,8	0,0	0,6	< 0,05	0,24	< 0,05	< 0,05	55,60	1,0	< 5%
Fate Sees_179	77,6	0,0	0,0	0,0	0,0	12,3	0,0	0,0	0,0	0,15	< 0,05	< 0,05	< 0,05	< 0,05	100,45	98,2	99%
Fate Sees_180	34,5	33,9	3,5	0,0	124,3	0,0	9,1	0,0	0,0	0,23	< 0,05	< 0,05	< 0,05	< 0,05	95,00	36,1	97%
Fate Sees_181	30,4	84,3	0,0	9,7	8,2	0,0	0,0	0,0	0,0	0,3	< 0,05	< 0,05	< 0,05	< 0,05	232,40	33,5	97%
Fate Sees_182	18,4	51,1	0,0	0,0	19,1	13,4	28,7	0,0	0,0	0,15	< 0,05	< 0,05	< 0,05	< 0,05	207,55	21,3	95%
Fate Sees_183	0,0	6,6	0,0	30,7	0,0	0,0	0,0	0,0	0,0	1,1	< 0,05	0,39	0,16	0,36	789,40	78,8	99%
Fate Sees_184	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	2,6	< 0,05	< 0,05	0,09	< 0,05	204,20	175,9	99%
Fate Sees_189	0,0	0,0	6,3	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	2,50	1,6	39%
Fate Sees_190	8,2	0,0	0,0	22,7	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	0,06	< 0,05	0,17	92,00	47,6	98%
Fate Sees_191	9,0	7,1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	33,90	18,8	95%
Fate Sees_193	0,0	3,2	0,0	0,0	0,0	0,0	0,0	57,7	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	10,65	13,8	93%
Fate Sees_194	35,3	11,9	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,38	0,07	< 0,05	< 0,05	14	66,40	83,3	99%

Fate Sees_195	0,0	123,1	0,0	227,8	17,0	0,0	0,0	0,0	0,0	0,87	0,65	< 0,05	0,31	0,22	32,55	3,6	72%
Fate Sees_196	0,0	33,7	0,0	99,5	6,5	0,0	0,0	0,0	0,0	0,77	< 0,05	< 0,05	< 0,05	0,72	3,00	2,0	49%
Fate Sees_198	32,8	0,0	0,0	57,7	0,0	0,0	61,7	0,0	0,0	0,17	< 0,05	0,09	0,1	< 0,05	20,95	12,9	92%
Fate Sees_199	34,0	14,1	0,0	25,1	30,1	0,0	0,0	0,0	0,0	1,7	< 0,05	0,55	1,2	< 0,05	118,20	70,0	99%
Fate Sees_202	81,3	29,1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,29	< 0,05	< 0,05	< 0,05	0,3	58,15	274,7	100%
Fate Sees_203	56,5	0,0	4,2	0,0	37,5	56,1	0,0	41,6	10,2	0,06	< 0,05	12	6,1	< 0,05	24,70	21,7	95%
Fate Sees_204	153,0	7,2	3,5	26,9	15,0	0,0	0,0	0,0	0,0	0,42	7,7	< 0,05	0,63	< 0,05	274,65	418,3	100%
Fate Sees_205	127,8	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,1	< 0,05	< 0,05	0,8	< 0,05	25,05	4,9	79%
Fate Sees_208	1,0	286,0	0,0	0,0	0,0	0,0	7,6	0,0	0,0	8,4	< 0,05	< 0,05	< 0,05	5,9	19,25	22,3	96%
Fate Sees_209	10,0	3618,5	0,0	238,2	0,0	0,0	30,7	0,0	0,0	< 0,05	< 0,05	< 0,05	0,15	0,23	33,60	12,2	92%
Fate Sees_210	92,6	657,0	0,0	0,0	0,0	72,0	36,3	110,6	4,7	< 0,05	< 0,05	< 0,05	< 0,05	2	3,50	1,2	17%
Fate Sees_211	9,0	74,2	1,1	0,0	9,1	34,1	0,0	98,6	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_214	4,3	1,1	2,3	0,0	28,3	36,6	7,6	11,7	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_226	2,2	1091,7	4,0	0,0	0,0	44,9	3,3	0,0	0,0	0,08	0,33	0,33	0,33	0,45	230,85	1,2	15%
Fate Sees_227	0,0	0,0	5,4	20,5	0,0	0,0	0,0	0,0	0,0	0,11	< 0,05	< 0,05	< 0,05	< 0,05	16,10	6,3	84%
Fate Sees_228	0,0	0,0	23,7	18,4	0,0	74,6	5,8	0,0	0,0	< 0,05	0,29	< 0,05	< 0,05	6,6	16,75	14,7	93%
Fate Sees_229	0,0	0,0	1,3	0,0	0,0	0,0	0,0	0,0	0,0	0,64	0,22	< 0,05	< 0,05	1,4	46,55	15,7	94%
Fate Sees_233	10,8	94,9	0,0	71,7	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	0,09	< 0,05	0,09	121,95	14,5	93%
Fate Sees_235	3,3	222,2	0,0	0,0	0,0	11,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	0,09	43,65	16,0	94%
Fate Sees_236	23,2	507,9	2,4	0,0	0,0	0,0	0,0	0,0	0,0	0,64	0,23	< 0,05	< 0,05	1,3	210,65	76,5	99%
Fate Sees_239	11,5	45,3	109,5	50,5	0,0	0,0	0,0	0,0	0,0	< 0,05	0,26	< 0,05	< 0,05	< 0,05	90,05	9,0	89%
Fate Sees_242	2,8	148,1	20,5	61,1	0,0	21,7	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	1	222,95	25,6	96%
Fate Sees_243	4,8	60,3	5,5	30,1	10,5	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	237,40	178,4	99%
Fate Sees_245	0,0	0,0	36,2	0,0	0,0	71,1	0,0	11,2	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	227,20	54,9	98%
Fate Sees_247	30,3	956,4	0,0	109,3	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	84,10	34,2	97%
Fate Sees_248	2,5	67,7	3,4	0,0	3,8	0,0	0,0	0,0	0,0	0,18	< 0,05	< 0,05	< 0,05	0,64	114,65	59,6	98%
Fate Sees_250	97,6	31,5	0,0	29,1	19,0	14,8	44,8	0,0	0,0	0,74	0,08	0,81	< 0,05	< 0,05	441,05	77,4	99%
Fate Sees_252																	
Fate Sees_253	0,0	0,0	0,0	197,2	20,1	21,9	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	15,70	1,0	< 5%
Fate Sees_254	103,0	500,0	0,0	117,8	0,0	54,8	26,2	0,0	0,0								
Fate Sees_256	27,9	0,0	0,0	13,2	0,0	0,0	0,0	0,0	0,0	1,6	< 0,05	< 0,05	< 0,05	< 0,05	58,70	5,6	82%
Fate Sees_257	9,7	805,8	0,0	67,1	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	145,60	6,5	85%
Fate Sees_258	3,7	429,4	3,4	0,0	0,0	0,0	0,0	0,0	0,0	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	304,45	48,5	98%
Fate Sees_259	78,6	975,9	5,7	208,4	0,0	10,5	0,0	0,0	0,0	2,9	< 0,05	< 0,05	< 0,05	150	49,50	1,0	< 5%
Fate Sees_260	87,6	79,9	7,4	0,0	0,0	25,9	0,0	7,3	8,9								
Fate Sees_261	9,6	58,1	12,8	0,0	0,0	139,3	5,2	54,4	0,0								
Fate Sees_262	7,0	46,4	3,8	0,0	3,6	30,2	0,0	0,0	0,0								
Fate Sees_263	2,5	50,9	16,1	0,0	0,0	27,5	0,0	121,9	0,0								
Fate Sees_264	40,6	67,6	8,8	0,0	3,1	5,4	2,1	0,0	0,0								
Fate Sees_265	0,0	1,4	0,0	17,3	0,0	192,8	0,0	13,2	0,0								
Fate Sees_266	10,9	276,7	11,9	0,0	0,0	0,0	0,0	0,0	0,0								
Fate Sees_267	15,7	107,1	2,4	0,0	0,0	5,6	63,4	0,0	0,0								
Fate Sees_268	75,6	160,5	54,6	0,0	6,1	54,9	2,7	28,4	0,0								

Annex 4: Results; Lab: JRC-IES. "0.0" means below LOQ.

Sample	Benzotriazole [µg/l]	Methylbenzo-triazole [µg/l]	DEET	Caffeine	Carbamazepine	Sulfamethoxazole	Diuron	Terbutylazine	DET	Atrazine	Isoproturon	Diazinon	DEA	Simazine	Metolachlor	Carbaryl	Linuron	Hexazinon	Chlortoluron	Methabenzthiazuron
LOQ [ng/l]	0.04	0.04	1	5	1	1	1	1	3	1	1	1	3	5	3	3	5	2	3	3
Fate Sees_136	0.85	1.76	88	2541	275	115	22	4	0.0	0.0	0.0	5	0.0	0.0	6	0.0	0.0	0.0	0.0	0.0
Fate Sees_137	1.49	1.37	629	169	1443	94	12	0.0	0.0	2	0.0	366	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_138	0.60	1.64	67	111	487	41	3	0.0	0.0	2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_139	4.22	4.43	199	3002	1656	174	368	7	0.0	4	270	16	0.0	0.0	8	0.0	0.0	0.0	0.0	0.0
Fate Sees_140	1.97	1.46	44	93	1136	131	10	75	126	2	1	2	0.0	5	73	0.0	0.0	0.0	0.0	0.0
Fate Sees_141	0.16	0.27	478	73	2177	4	0.0	0.0	2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_142	0.19	0.31	12	14	4609	0.0	0.0	0.0	1	0.0	0.0	0.0	0.0	0.0	27	0.0	0.0	0.0	0.0	0.0
Fate Sees_143	3.92	2.56	1050	9	538	119	107	38	194	8	1	2	45	0.0	14	0.0	0.0	0.0	0.0	0.0
Fate Sees_144	1.10	2.90	148	8	1021	82	7	63	0.0	2	1	5	55	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_145	2.91	1.50	275	27	1481	180	49	13	59	5	3	5	0.0	0.0	0.0	0.0	49	0.0	0.0	0.0
Fate Sees_146	2.62	1.69	257	7	979	257	44	8	87	9	12	5	0.0	0.0	0.0	8	0.0	0.0	0.0	0.0
Fate Sees_147	4.99	2.21	58	19	906	623	26	51	125	21	10	4	75	36	0.0	3	0.0	0.0	0.0	0.0
Fate Sees_148	2.77	3.04	265	0.0	1051	100	11	39	17	14	55	4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_149	6.53	3.91	959	28	879	453	346	59	302	6	6	4	54	0.0	55	14	0.0	0.0	0.0	0.0
Fate Sees_150	0.52	1.38	355	0.0	81	0.0	0.0	0.0	0.0	21	2	5	0.0	0.0	0.0	0.0	0.0	42	0.0	0.0
Fate Sees_151	0.17	7.26	23	66	0.0	0.0	0.0	127	0.0	37	3	3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_152	1.04	18.48	11	1518	0.0	0.0	1077	29	0.0	19	2	4	0.0	0.0	15	0.0	0.0	0.0	184	0.0
Fate Sees_153	0.00	0.74	8	85	0.0	0.0	22	0.0	28	4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_155	0.38	0.00	55	0.0	281	0.0	0.0	10	88	0.0	0.0	0.0	0.0	0.0	21	0.0	0.0	0.0	0.0	0.0
Fate Sees_156	0.23	1.96	26	30	0.0	0.0	0.0	53	54	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_157	0.00	0.01	8	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_158	2.18	14.49	158	126	373	0.0	60	115	78	11	35	6	0.0	0.0	112	0.0	59	0.0	0.0	0.0
Fate Sees_159	0.65	24.32	12	123	0.0	0.0	0.0	25	0.0	0.0	4	0.0	0.0	0.0	17	0.0	0.0	0.0	0.0	0.0
Fate Sees_160	3.80	3.83	8	838	26	0.0	0.0	3	121	0.0	0.0	0.0	89	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_161	221.17	0.29	25	760	0.0	0.0	0.0	10	0.0	8	0.0	0.0	155	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_162	0.04	0.29	15	0.0	0.0	0.0	0.0	13	0.0	0.0	0.0	3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	3
Fate Sees_163	0.00	0.70	17	11	29	8	0.0	122	0.0	2	1	2	21	141	0.0	0.0	17	0.0	27	3
Fate Sees_164	0.05	0.04	5	11	0.0	0.0	3	5	28	2	0.0	0.0	68	0.0	0.0	0.0	0.0	0.0	0.0	18
Fate Sees_165	3.17	0.78	1346	144	1146	46	47	55	0.0	7	0.0	2	0.0	0.0	19	0.0	0.0	0.0	0.0	0.0
Fate Sees_166	2.45	1.32	187	112	1282	288	82	38	32	6	2	17	0.0	28	49	0.0	0.0	0.0	0.0	0.0
Fate Sees_167	7.52	2.55	74	692	1624	86	53	0.0	67	3	0.0	30	0.0	48	0.0	0.0	0.0	6	0.0	0.0
Fate Sees_168	2.55	2.52	393	32	2209	87	49	33	0.0	3	4	3	0.0	0.0	37	0.0	19	5	9	0.0
Fate Sees_169	14.06	9.85	1858	112	1323	66	39	19	58	5	3	3	0.0	60	11	0.0	0.0	0.0	0.0	0.0
Fate Sees_170	4.54	2.81	331	35	692	328	10	1	6	7	18	7	45	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_171	3.39	2.05	15791	387	891	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	6	0.0	0.0
Fate Sees_175	3.77	2.82	3932	22	709	28	0.0	23	48	20	0.0	0.0	0.0	0.0	37	0.0	0.0	4	0.0	0.0
Fate Sees_177	2.35	2.39	104	100	815	105	0.0	0.0	0.0	4	2	8	0.0	13	0.0	0.0	0.0	2	5	0.0
Fate Sees_179	14.81	1.80	52	13	541	113	4	0.0	0.0	4	2	4	94	0.0	10	0.0	0.0	0.0	11	0.0
Fate Sees_180	4.22	4.57	2913	26	1144	21	14	0.0	0.0	0.0	0.0	0.0	58	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_181	2.54	1.88	1058	53	718	86	8	0.0	48	0.0	0.0	2	22	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_182	5.33	3.76	1091	75	666	33	0.0	0.0	143	0.0	0.0	0.0	0.0	80	0.0	0.0	0.0	4	0.0	0.0
Fate Sees_183	2.96	3.18	243	13	690	46	10	0.0	13	0.0	2	0.0	25	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_184	0.96	1.14	953	0.0	419	70	7	0.0	422	5	0.0	4	0.0	113	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_189	0.19	0.06	374	6	0.0	0.0	0.0	0.0	98	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Fate Sees_190	4.05	1.39	650	46	84	884	17	6	0.0	2	39	391	0.0	0.0	0.0	0.0	0.0	0.0	34	0.0

Fate Sees_191	9,00	2,32	73	7	230	591	46	19	0,0	11	50	23	59	0,0	14	0,0	0,0	0,0	0,0	0,0
Fate Sees_193	6,18	3,19	111	34	604	210	21	4	56	6	19	58	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_194	7,46	1,53	2	53	340	218	8	7	0,0	4	14	22	30	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_195	12,94	3,59	510	67	648	433	16	38	21	5	6	40	0,0	16	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_196	2,55	3,23	1098	45	837	65	66	0,0	0,0	4	0,0	11	0,0	106	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_198	7,18	5,51	949	196	1378	90	97	0,0	4	0,0	0,0	9	0,0	33	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_199	9,05	3,23	237	12	856	22	22	14	101	0,0	11	25	35	0,0	9	0,0	0,0	0,0	0,0	0,0
Fate Sees_202	14,67	3,34	285	12	606	35	21	19	0,0	2	4	9	0,0	0,0	25	0,0	0,0	0,0	0,0	0,0
Fate Sees_203	16,11	2,39	502	14	734	22	19	11	20	2	21	5	0,0	0,0	50	7	3265	0,0	0,0	0,0
Fate Sees_204	6,08	1,97	120	25	578	60	23	14	88	3	23	5	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_205	11,07	4,17	167	518	969	44	17	7	0,0	4	14	12	0,0	0,0	0,0	0,0	0,0	13	0,0	0,0
Fate Sees_208	14,31	3,06	117	12	2678	15	33	3	0,0	26	4	13	0,0	0,0	10	0,0	0,0	0,0	0,0	0,0
Fate Sees_209	2,68	2,10	253	14	303	141	192	994	493	3	1	12	0,0	142	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_210	4,62	7,85	192	30	317	38	1426	733	535	0,0	4	33	0,0	30	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_211	3,88	2,46	88	18	161	645	517	2411	1531	0,0	2	14	0,0	689	0,0	81	0,0	0,0	0,0	0,0
Fate Sees_214	0,75	1,35	49	64	1142	59	13	0,0	0,0	18	0	5	131	26	55	0,0	0,0	3	0,0	0,0
Fate Sees_226	0,87	0,43	34	9	155	52	2	2	79	1	0,0	3	51	15	5	5	0,0	0,0	0,0	0,0
Fate Sees_227	1,15	0,70	358	162	425	30	5	0,0	314	2	0,0	366	0,0	107	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_228	0,58	0,38	19	20	236	194	0,0	0,0	122	0,0	0,0	5	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_229	1,90	2,13	1798	82	1233	223	4	2	11	3	2	28	29	40	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_233	0,43	0,47	3	20	781	106	38	1	0,0	0,0	0,0	16	0,0	151	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_235	4,20	2,85	567	32	1254	152	7	2	119	0,0	0,0	6	0,0	18	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_236	2,98	2,06	139	37	747	70	6	5	11	0,0	0,0	18	0,0	353	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_239	9,71	2,60	409	65	1392	398	4	2	0,0	0,0	3	5	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_242	5,19	3,32	490	47	965	187	13	299	48	0,0	1	6	15	12	289	0,0	199	0,0	0,0	16
Fate Sees_243	4,99	2,64	560	152	1774	190	39	85	130	0,0	15	15	0,0	0,0	53	0,0	0,0	0,0	0,0	0,0
Fate Sees_245	6,60	3,96	518	34	1176	152	30	95	15	2	27	16	0,0	0,0	28	0,0	0,0	0,0	0,0	0,0
Fate Sees_247	0,98	0,34	76	54	690	3	0,0	0,0	0,0	4	0,0	1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_248	10,86	6,48	1575	42	1549	405	43	17	0,0	0,0	0,0	30	41	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_250	3,71	2,37	289	19	1620	167	11	0,0	47	2	0,0	5	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_252	18,43	8,65	491	0,0	1979	242	22	520	63	0,0	201	4	40	0,0	18	0,0	0,0	0,0	0,0	0,0
Fate Sees_253																				
Fate Sees_254	0,10	0,82	9658	254	13	0,0	0,0	2	174	2	0,0	0,0	0,0	66	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_256	0,88	1,04	1	1148	972	62	0,0	0,0	0,0	0,0	0	0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_257	6,59	2,14	765	144	1539	1147	0,0	67	18	3	0,0	3	0,0	0,0	113	0,0	0,0	0,0	0,0	0,0
Fate Sees_258	4,23	2,46	544	18	1318	170	17	1704	0,0	0,0	0,0	105	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_259	4,81	1,94	410	21	1119	229	18	22	0,0	3	2	13	0,0	0,0	0,0	0,0	0,0	0,0	0,0	3
Fate Sees_260	4,64	2,63	248	28	885	756	120	20	0,0	0,0	0,0	70	0,0	0,0	20	0,0	0,0	0,0	0,0	0,0
Fate Sees_261	0,90	0,85	100	16	856	34	3	1	0,0	2	0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_262	0,31	1,06	58	71	767	46	14	0,0	0,0	3	0,0	1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_263	1,04	1,48	276	99	1046	180	75	0,0	0,0	1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_264	0,27	1,44	9	12	508	0,0	14	1	0,0	1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	6	0,0	0,0
Fate Sees_265	0,50	0,83	39	77	444	42	5	1	24	3	1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_266	0,68	0,86	67	12	757	21	2	0	0,0	2	0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_267	0,64	1,24	99	1588	900	68	6	0,0	14	2	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
Fate Sees_268	0,51	1,90	95	265	653	67	6	0,0	0,0	1	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0

Annex 5: Results for veterinary antibiotics; Lab: VITO, Belgium. Florfenicol < 100 ng/l, Tylosine < 50 ng/l, and Fenbendazole < 150 ng/l in all samples.

Sample	Oxytetracycline	Doxycycline	Penicilline V	Penicilline G	Amoxilline	Ampicilline	Tilmicosine	Clindamycine	Lincomycine	Tiamuline	Sulfamethoxazole	Sulfadiazine	Sulfadoxine	Trimethoprim	Flumequine	Enrofloxacin	Ciprofloxacin	Levamisole
LOQ [ng/l]	30	50	50	50	25	25	50	10	15	20	20	30	50	10	20	20	15	15
Fate Sees_136	<30	<50	<50	<50	<25	<25	<50	<10	<15	44	<20	<30	<50	<10	<20	<20	62	24
Fate Sees_137	<30	<50	<50	<50	<30	<25	<50	<10	<15	<20	164	<30	<50	37	<20	<20	52	<15
Fate Sees_138	<30	<50	<50	<50	<25	<25	<50	<15	<50		<20	<30	<50	<10	<20	<20	<15	<15
Fate Sees_139																		
Fate Sees_140	<30	<50	<50	<50	<25	<25	<50	<15	<50		32	<30	<50	24	<20	<20	<15	<15
Fate Sees_141																		
Fate Sees_142	<30	<50	<50	-	<25	<25	<50	<15	<50		<20	<30	<50	11	<20	<20	<15	<15
Fate Sees_143	<30	<50	95	<50	<25	<25	<50	53	<15	<20	301	<30	<50	436	<20	<20	55	<15
Fate Sees_144	<30	<50	101	<50	<25	<25	<50	133	<15	<20	171	<30	<50	313	<20	<20	58	<15
Fate Sees_145	<30	<50	59	-	<25	<25	<50	134	53	<20	343	<30	<50	375	<20	<20	95	<15
Fate Sees_146	<30	<50	79	<50	<25	<25	<50	89	21	<20	434	<30	<50	244	<20	<20	60	109
Fate Sees_147	<30	<50	<50	<50	<25	<25	<50	53	317	<20	1690	<30	<50	233	<20	<20	74	<15
Fate Sees_148	<30	<50	<50	<50	<25	<25	<50	132	198	<20	47	<30	<50	25	<20	<20	96	<15
Fate Sees_149	<30	<50	<50	<50	<25	<25	<50	211	<15	<20	616	<30	<50	552	<20	<20	61	<15
Fate Sees_150																		
Fate Sees_151																		
Fate Sees_152																		
Fate Sees_153																		
Fate Sees_155																		
Fate Sees_156																		
Fate Sees_157																		
Fate Sees_158																		
Fate Sees_159																		
Fate Sees_160																		
Fate Sees_161																		
Fate Sees_162																		
Fate Sees_163																		
Fate Sees_164																		
Fate Sees_165																		
Fate Sees_166																		
Fate Sees_167																		
Fate Sees_168																		
Fate Sees_169	<30	<50	101	<50	<25	<25	<50	19	<15	<20	633	105	<50	723	<20	<20	235	92
Fate Sees_170																		
Fate Sees_171																		
Fate Sees_175																		
Fate Sees_177																		
Fate Sees_179																		
Fate Sees_180																		
Fate Sees_181																		
Fate Sees_182																		
Fate Sees_183																		
Fate Sees_184																		
Fate Sees_189																		

Fate Sees_190																		
Fate Sees_191																		
Fate Sees_193																		
Fate Sees_194	<30	<50	62	<50	<25	<25	<50	125	<15	<20	448	<30	<50	310	<20	<20	195	112
Fate Sees_195	<30	<50	<50	<50	<25	<25	<50	37	<15	<20	96	<30	<50	561	<20	<20	124	19
Fate Sees_196	<30	<50	<50	<50	<25	<25	<50	39	<15	<20	81	<30	<50	440	<20	<20	217	57
Fate Sees_198																		
Fate Sees_199																		
Fate Sees_202																		
Fate Sees_203	<30	<50	<50	<50	<25	<25	<50	129	104	<20	109	<30	<50	176	<20	<20	104	<15
Fate Sees_204	<30	<50	<50	<50	<25	<25	<50	277	<15	<20	60	<30	<50	180	<20	<20	117	<15
Fate Sees_205	<30	<50	<50	<50	<25	<25	<50	147	22	<20	31	<30	<50	208	<20	<20	115	<15
Fate Sees_208	<30	<50	97	--	<25	<25	<50	10	<15	<20	195	<30	<50	75	<20	<20	96	77
Fate Sees_209	<30	<50	86	<50	<25	<25	<50	79	<15	29	<20	<30	<50	47	<20	<20	265	149
Fate Sees_210	<30	<50	122	<50	<25	<25	<50	187	29	<20	1183	<30	<50	800	<20	<20	189	<15
Fate Sees_211																		
Fate Sees_214																		
Fate Sees_226	<30	<50	59	<50	<25	<25	<50	17	28	24	101	<30	<50	76	<20	<20	59	<15
Fate Sees_227	<30	<50	<50	<50	<25	<25	<50	17	17	<20	388	<30	<50	149	<20	<20	91	<15
Fate Sees_228	<30	<50	<50	<50	<25	<25	93	26	128	<20	228	<30	<50	141	<20	<20	87	<15
Fate Sees_229	<30	<50	<50	<50	<25	<25	<50	<10	<15	<20	164	<30	<50	120	26	<20	49	42
Fate Sees_233																		
Fate Sees_235																		
Fate Sees_236	<30	<50	<50	<50	<25	<25	<50	91	<15	<20	564	<30	<50	207	<20	<20	71	199
Fate Sees_239																		
Fate Sees_242																		
Fate Sees_243																		
Fate Sees_245																		
Fate Sees_247																		
Fate Sees_248	<30	<50	<50	<50	<25	<25	<50	109	18	<20	202	<30	<50	204	<20	<20	77	340
Fate Sees_250																		
Fate Sees_252																		
Fate Sees_253																		
Fate Sees_254	<30	<50	<50	<50	<25	<25	<50	<15	<50		122	<30	<50	104	<20	<20	119	<15
Fate Sees_256																		
Fate Sees_257																		
Fate Sees_258																		
Fate Sees_259	<30	<50	<50	<50	<25	<25	<50	<15	<50		<20	<30	<50	99	<20	<20	66	<15
Fate Sees_260																		
Fate Sees_261																		
Fate Sees_262																		
Fate Sees_263																		
Fate Sees_264																		
Fate Sees_265																		
Fate Sees_266																		
Fate Sees_267																		
Fate Sees_268																		

Annex 6: Results for siloxanes and musk fragrances; Lab: UBA, Austria; n.n. means < LOQ.

Sample	Decamethylcyclopentasiloxan (D5)	Decamethyltetrasiloxan (MD2M)	Dodecamethylcyclohexasiloxan (D6)	Dodecamethylpentasiloxan (MD3M)	Octamethylcyclotetrasiloxan (D4)	Octamethyltrisiloxan (MDM)	Cashmeran	Celestolid	Galaxolid	Phantolid	Tonalid	Traesolid
LOQ [$\mu\text{g/l}$]	0,055	0,01	0,035	0,01	0,02	0,005	0,005	0,015	0,015	0,005	0,015	0,005
Fate Sees_136	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_137	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,013	n.n.	0,038	n.n.	n.n.	n.n.
Fate Sees_138	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_139	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,13	n.n.	0,19	n.n.	< 0,030	n.n.
Fate Sees_140	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_141	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,19	n.n.	0,26	n.n.	n.n.	< 0,010
Fate Sees_142	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_143	n.n.	n.n.	n.n.	n.n.	< 0,040	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_144	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_145	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,016	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_146	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_147	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_148	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_149	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_150	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,010	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_151	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,010	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_152	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,017	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_153	0,28	n.n.	0,68	n.n.	0,11	n.n.	0,062	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_155	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_156	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_157	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_158	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,030	n.n.
Fate Sees_159	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_160	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_161	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,030	n.n.
Fate Sees_162	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,010	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_163	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_164	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,073	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_165	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,035	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_166	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,046	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_167	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,025	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_168	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,021	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_169	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_170	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,075	n.n.	< 0,030	n.n.	n.n.	n.n.

Fate Sees_171	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,031	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_175	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,018	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_177	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_179	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,055	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_180	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,024	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_181	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Fate Sees_182	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,033	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_183	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,06	n.n.	0,036	n.n.	n.n.	n.n.
Fate Sees_184	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,022	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_189	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_190	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,084	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_191	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,036	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_193	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,02	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_194	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,014	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_195	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,22	n.n.	0,16	n.n.	< 0,030	n.n.
Fate Sees_196	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,18	n.n.	0,1	n.n.	n.n.	n.n.
Fate Sees_198	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_199	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,031	n.n.	0,032	n.n.	n.n.	n.n.
Fate Sees_202	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_203	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_204	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_205	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,010	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_208	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,010	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_209	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,021	n.n.	0,091	n.n.	n.n.	n.n.
Fate Sees_210	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,03	n.n.	n.n.	n.n.
Fate Sees_211												
Fate Sees_214												
Fate Sees_226	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,048	n.n.	n.n.	n.n.
Fate Sees_227	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_228	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_229	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_233	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,48	n.n.	0,14	n.n.	n.n.	n.n.
Fate Sees_235	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,11	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_236	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,053	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_239	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,07	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_242	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,071	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_243	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,098	n.n.	n.n.	n.n.	< 0,010	n.n.
Fate Sees_245	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,074	n.n.	0,099	n.n.	n.n.	n.n.
Fate Sees_247	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,095	n.n.	< 0,030	n.n.	n.n.	n.n.
Fate Sees_248	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,13	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_250	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,017	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_252	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,056	n.n.	0,072	n.n.	n.n.	n.n.
Fate Sees_253	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,018	n.n.	0,032	n.n.	n.n.	n.n.
Fate Sees_254	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,2	n.n.	0,13	n.n.	0,077	n.n.
Fate Sees_256	n.n.	n.n.	n.n.	n.n.	< 0,040	n.n.	0,012	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_257	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,11	n.n.	n.n.	n.n.	n.n.	n.n.
Fate Sees_258	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,067	n.n.	0,032	n.n.	n.n.	n.n.
Fate Sees_259	n.n.	n.n.	n.n.	n.n.	n.n.	n.n.	0,095	n.n.	0,088	n.n.	n.n.	n.n.
Fate Sees_260												
Fate Sees_261												
Fate Sees_262												
Fate Sees_263												
Fate Sees_264												
Fate Sees_265												
Fate Sees_266												
Fate Sees_267												
Fate Sees_268												

Annex 7: Results for organophosphate ester flame retardants; Lab: UMEÅ University, Sweden.

Sample	Tri-iso-butyphosphate	Tributyphosphate	Tris(2-chloroethyl)phosphate	Tris(2-chloroisopropyl)phosphate	Tris(1,3-dichloro-2-propyl)phosphate	Tris(2-butoxyethyl)phosphate	Triphenylphosphate	2-Ethylhexyldiphenylphosphate	Tris(2-ethylhexyl)phosphate	Tricresylphosphate
LOQ [ng/l]	1	1	1	1	1	1	1	1	1	1
Fate Sees_136	110	330	140	970	73	56	8,7	2,4	<LOQ	<LOQ
Fate Sees_137	150	190	200	1600	190	190	12	2,7	<LOQ	<LOQ
Fate Sees_138	130	89	120	810	420	78	11	3,4	<LOQ	<LOQ
Fate Sees_139										
Fate Sees_140	170	140	2400	1600	610	89	53	9,4	<LOQ	<LOQ
Fate Sees_141										
Fate Sees_142	160	250	400	4300	860	470	180	44	<LOQ	<LOQ
Fate Sees_143	120	260	75	1000	470	20	130	3,7	<LOQ	<LOQ
Fate Sees_144	85	140	270	1400	170	47	34	6,3	<LOQ	<LOQ
Fate Sees_145	110	110	62	700	120	110	18	11	<LOQ	<LOQ
Fate Sees_146	54	64	33	400	120	130	4,3	5,6	<LOQ	<LOQ
Fate Sees_147	76	73	66	940	210	54	21	5,4	<LOQ	<LOQ
Fate Sees_148	75	420	21	900	5,0	230	3,2	13	<LOQ	<LOQ
Fate Sees_149	170	220	90	890	260	230	34	6,6	<LOQ	<LOQ
Fate Sees_150	82	61	58	21000	2,8	9300	11	<LOQ	<LOQ	<LOQ
Fate Sees_151	130	910	160	710	21	670	17	7,7	<LOQ	<LOQ
Fate Sees_152	130	68	22	360	24	490	12	9,8	<LOQ	<LOQ
Fate Sees_153	280	760	36	60	10	1400	610	<LOQ	<LOQ	<LOQ
Fate Sees_155	130	100	120	5800	9,9	190	71	<LOQ	<LOQ	<LOQ
Fate Sees_156	78	83	5,6	40	16	18	12	9,7	<LOQ	<LOQ
Fate Sees_157	87	52	27	170	46	340	7,6	10	<LOQ	<LOQ
Fate Sees_158	79	100	120	450	17	29	51	20	<LOQ	<LOQ
Fate Sees_159	93	58	21	60	22	190	19	7,7	<LOQ	<LOQ
Fate Sees_160	130	140	37	160	15	52	37	21	<LOQ	<LOQ
Fate Sees_161	160	360	200	310	5,6	11	20	6,5	<LOQ	<LOQ
Fate Sees_162	140	74	17	500	16	14	12	5,7	<LOQ	<LOQ
Fate Sees_163	110	480	18	530	210	23	20	5,4	<LOQ	<LOQ
Fate Sees_164	140	210	270	4400	430	650	75	35	<LOQ	<LOQ
Fate Sees_165	64	89	90	410	480	92	10	<LOQ	<LOQ	<LOQ
Fate Sees_166	100	66	200	2400	360	70	21	5,2	<LOQ	<LOQ
Fate Sees_167	78	48	310	1900	790	29	150	42	<LOQ	<LOQ

Fate Sees_168	110	50	47	1200	26	60	19	4,3	<LOQ	<LOQ
Fate Sees_169	150	390	63	1300	330	410	16	5,5	<LOQ	<LOQ
Fate Sees_170	160	290	90	970	150	7900	49	3,2	<LOQ	<LOQ
Fate Sees_171	72	510	18	120	96	7500	16	7,2	<LOQ	<LOQ
Fate Sees_175	110	120	47	490	68	27	17	7,5	<LOQ	<LOQ
Fate Sees_177	100	200	36	400	46	33	6,5	2,3	<LOQ	<LOQ
Fate Sees_179	130	190	75	320	170	89	16	1,8	<LOQ	<LOQ
Fate Sees_180	88	310	110	2000	170	200	6,2	1,0	<LOQ	<LOQ
Fate Sees_181	170	400	85	400	190	4600	15	7,7	<LOQ	<LOQ
Fate Sees_182	170	200	73	370	130	1200	13	5,8	<LOQ	<LOQ
Fate Sees_183	230	890	97	690	90	75	14	32	<LOQ	<LOQ
Fate Sees_184	100	440	120	630	63	10	6,4	6,0	<LOQ	<LOQ
Fate Sees_189	83	110	50	380	28	340	8,4	9,3	<LOQ	<LOQ
Fate Sees_190	120	110	48	610	50	37	24	6,5	<LOQ	<LOQ
Fate Sees_191	120	81	33	510	58	240	17	9,2	<LOQ	<LOQ
Fate Sees_193	100	91	71	1200	57	270	27	4,9	<LOQ	<LOQ
Fate Sees_194	56	310	43	1200	110	1700	51	20	<LOQ	<LOQ
Fate Sees_195	150	180	310	10000	450	84	46	11	<LOQ	<LOQ
Fate Sees_196	140	81	110	990	83	8,5	31	4,3	<LOQ	<LOQ
Fate Sees_198	92	440	200	1100	580	43000	120	1000	<LOQ	<LOQ
Fate Sees_199	340	860	56	320	100	2700	84	5400	<LOQ	<LOQ
Fate Sees_202	260	370	190	270	86	62	16	26	<LOQ	<LOQ
Fate Sees_203	57	120	21	360	58	110	32	11	<LOQ	<LOQ
Fate Sees_204	77	390	50	290	140	230	6,9	43	<LOQ	<LOQ
Fate Sees_205	200	590	200	1100	300	20	33	12	<LOQ	<LOQ
Fate Sees_208	46	230	52	950	120	410	7,4	5,0	<LOQ	<LOQ
Fate Sees_209	580	1100	790	3300	480	1300	110	210	<LOQ	<LOQ
Fate Sees_210	320	330	240	2100	120	42	19	3,3	<LOQ	<LOQ
Fate Sees_211	8,3	8,5	11	150	35	12000	9,2	5,5	<LOQ	<LOQ
Fate Sees_214	8,2	3,7	23	220	46	5400	1,6	2,0	<LOQ	<LOQ
Fate Sees_226	130	260	17	130	52	77	8,5	2,2	<LOQ	<LOQ
Fate Sees_227	73	140	47	200	47	31	10	3,3	<LOQ	<LOQ
Fate Sees_228	220	220	32	440	6,0	72	51	16	<LOQ	<LOQ
Fate Sees_229	110	370	210	470	120	1700	16	3,6	<LOQ	<LOQ
Fate Sees_233	180	230	140	1400	130	180	66	8,6	<LOQ	<LOQ
Fate Sees_235	130	430	71	600	170	100	35	710	<LOQ	<LOQ
Fate Sees_236	140	570	88	1400	200	2100	16	11	<LOQ	<LOQ
Fate Sees_239	94	86	58	620	180	46	6,4	1,0	<LOQ	<LOQ
Fate Sees_242	260	730	170	1600	500	770	38	37	<LOQ	<LOQ
Fate Sees_243	130	750	180	1300	560	250	41	62	<LOQ	<LOQ
Fate Sees_245	140	140	270	2200	290	150	36	16	<LOQ	<LOQ
Fate Sees_247	240	560	120	1100	240	2000	31	46	<LOQ	<LOQ
Fate Sees_248	73	120	180	2100	370	1200	19	4,1	<LOQ	<LOQ
Fate Sees_250	290	1700	160	1100	620	130	45	64	<LOQ	<LOQ
Fate Sees_252	110	150	160	780	71	270	44	8,6	<LOQ	<LOQ
Fate Sees_253	150	340	7,3	54	150	46	15	19	<LOQ	<LOQ
Fate Sees_254	870	330	330	1600	500	13000	95	23	<LOQ	<LOQ
Fate Sees_256	61	130	70	420	26	29	3,2	1,2	<LOQ	<LOQ
Fate Sees_257	130	140	62	470	44	53	7,3	2,5	<LOQ	<LOQ
Fate Sees_258	190	160	110	710	190	56	12	<LOQ	<LOQ	<LOQ
Fate Sees_259	310	160	84	940	300	18	29	3,5	<LOQ	<LOQ
Fate Sees_260	5,0	6,2	11	92	28	13000	7,1	6,7	<LOQ	<LOQ
Fate Sees_261	6,0	7,4	23	370	190	9900	5,3	2,2	<LOQ	1,1
Fate Sees_262	11	10	1,5	25	9,5	2600	2,2	3,0	<LOQ	<LOQ
Fate Sees_263	5,8	3,4	17	98	50	1400	2,1	4,0	<LOQ	<LOQ
Fate Sees_264	1,7	10	4,5	38	12	6700	2,6	2,3	<LOQ	<LOQ
Fate Sees_265	5,1	7,5	27	170	83	19000	7,4	12	<LOQ	<LOQ
Fate Sees_266	7,2	5,5	26	200	64	3100	4,9	1,8	<LOQ	1,3
Fate Sees_267	6,4	7,5	16	150	35	10000	1,3	3,0	<LOQ	<LOQ
Fate Sees_268	3,8	9,1	6,5	90	29	4300	5,4	5,2	<LOQ	<LOQ

Annex 8: Results for pharmaceuticals; Lab: UMEÅ University, Sweden.

Sample	Alfuzosin	Alprazolam	Amitriptyline	Atorvastatin	Azelastine	Biperiden	Bisoprolol	Bromocriptin	Buprenorphin	Bupropion	Chlorthalidon	Chlorpromazine	Citalopram	Citalopram	Clemastine	Clomipramine	Clonazepam	Clotrimazol
LOQ [ng/l]	0,1	10	5	59	5	0,1	0,1	5	10	0,1	10	5	1	5	0,5	0,5	5	1
Fate Sees_136	0,3	<LOQ	<LOQ	<LOQ	<LOQ	0,1	2,2	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	7,3	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_137	0,6	<LOQ	<LOQ	<LOQ	<LOQ	0,1	11,1	<LOQ	<LOQ	0,6	<LOQ	<LOQ	<LOQ	5,8	<LOQ	<LOQ	<LOQ	1,7
Fate Sees_138	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	12,3	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,9	<LOQ	<LOQ
Fate Sees_139																		
Fate Sees_140	4,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	54,3	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	28,0	<LOQ	0,7	<LOQ	<LOQ
Fate Sees_141																		
Fate Sees_142	3,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	15,0	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	8,4	<LOQ	0,8	<LOQ	1,3
Fate Sees_143	3,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	17,1	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	44,4	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_144	3,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	18,1	<LOQ	<LOQ	1,4	<LOQ	<LOQ	<LOQ	74,4	<LOQ	<LOQ	<LOQ	2,6
Fate Sees_145	0,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	3,9	<LOQ	<LOQ	0,5	<LOQ	<LOQ	<LOQ	65,9	<LOQ	<LOQ	<LOQ	1,5
Fate Sees_146	0,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	8,2	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	21,1	<LOQ	<LOQ	<LOQ	2,2
Fate Sees_147	0,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	23,3	<LOQ	<LOQ	0,5	<LOQ	<LOQ	<LOQ	36,7	<LOQ	<LOQ	<LOQ	2,5
Fate Sees_148	0,5	<LOQ	<LOQ	<LOQ	<LOQ	0,1	9,5	<LOQ	<LOQ	0,6	<LOQ	<LOQ	<LOQ	19,1	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_149	1,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	36,4	<LOQ	<LOQ	1,9	<LOQ	<LOQ	1,3	48,9	<LOQ	<LOQ	<LOQ	1,1
Fate Sees_150	0,8	25,4	<LOQ	<LOQ	<LOQ	<LOQ	0,9	<LOQ	28,6	1,1	<LOQ	<LOQ	<LOQ	8,5	<LOQ	1,3	40,7	5,3
Fate Sees_151	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,3	0,5	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	9,8	<LOQ	0,9	12,2	<LOQ
Fate Sees_152	0,5	10,6	8,8	<LOQ	<LOQ	<LOQ	0,7	<LOQ	31,2	1,2	<LOQ	10,4	<LOQ	5,5	<LOQ	3,0	15,8	2,3
Fate Sees_153	<LOQ	20,5	<LOQ	<LOQ	<LOQ	2,3	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	10,9	<LOQ	2,6	<LOQ	<LOQ
Fate Sees_155	0,7	<LOQ	<LOQ	<LOQ	<LOQ	0,2	0,5	<LOQ	<LOQ	0,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_156	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_157	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1,4	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_158	0,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_159	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	7,7	<LOQ
Fate Sees_160	0,3	11,4	<LOQ	<LOQ	<LOQ	<LOQ	0,8	<LOQ	259,1	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_161	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_162	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_163	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_164	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	<LOQ	15,1	0,2	<LOQ	<LOQ	<LOQ	10,7	<LOQ	1,0	<LOQ	<LOQ
Fate Sees_165	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	13,3	<LOQ	<LOQ	0,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1,3	<LOQ	1,3
Fate Sees_166	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	64,3	<LOQ	<LOQ	0,9	<LOQ	<LOQ	<LOQ	14,7	<LOQ	<LOQ	<LOQ	1,2
Fate Sees_167	0,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	133,8	<LOQ	<LOQ	4,2	<LOQ	<LOQ	<LOQ	33,9	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_168	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	32,6	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	14,8	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_169	0,2	20,1	<LOQ	<LOQ	<LOQ	<LOQ	20,1	<LOQ	<LOQ	4,9	<LOQ	<LOQ	<LOQ	49,1	<LOQ	<LOQ	<LOQ	1,2
Fate Sees_170	13,1	10,8	<LOQ	72,9	<LOQ	<LOQ	423,4	<LOQ	12,6	0,3	<LOQ	<LOQ	<LOQ	188,6	<LOQ	<LOQ	<LOQ	2,0
Fate Sees_171	9,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	271,0	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	155,9	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_175	0,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	6,9	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_177	2,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	21,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	4,9
Fate Sees_179	8,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	257,6	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	87,3	<LOQ	<LOQ	<LOQ	1,4
Fate Sees_180	8,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	195,9	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	153,8	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_181	3,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	163,7	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	63,4	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_182	4,5	<LOQ	<LOQ	<LOQ	<LOQ	0,1	186,2	<LOQ	<LOQ	0,5	<LOQ	<LOQ	<LOQ	60,7	<LOQ	<LOQ	7,3	1,1

Fate Sees_183	20,5	<LOQ	<LOQ	<LOQ	<LOQ	0,3	6,7	<LOQ	<LOQ	0,9	<LOQ	<LOQ	<LOQ	48,1	<LOQ	0,5	<LOQ	1,4
Fate Sees_184	0,2	<LOQ	<LOQ	<LOQ	<LOQ	0,2	0,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	
Fate Sees_189	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	14,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	28,3	<LOQ	<LOQ	<LOQ	
Fate Sees_190	1,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	10,4	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	20,6	<LOQ	0,8	<LOQ	
Fate Sees_191	1,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	9,8	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	32,0	<LOQ	<LOQ	<LOQ	
Fate Sees_193	0,5	<LOQ	<LOQ	<LOQ	<LOQ	0,2	25,2	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	21,6	<LOQ	<LOQ	<LOQ	
Fate Sees_194	3,4	<LOQ	14,6	<LOQ	<LOQ	0,1	47,3	<LOQ	<LOQ	2,7	<LOQ	<LOQ	<LOQ	56,2	<LOQ	<LOQ	<LOQ	
Fate Sees_195	1,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	4,8	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	34,4	<LOQ	<LOQ	1,2	
Fate Sees_196	2,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	12,8	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	29,7	<LOQ	<LOQ	<LOQ	
Fate Sees_198	0,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	1,9	<LOQ	<LOQ	<LOQ	39,6	<LOQ	<LOQ	<LOQ	
Fate Sees_199	1,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	38,3	<LOQ	<LOQ	2,0	<LOQ	<LOQ	<LOQ	61,0	<LOQ	<LOQ	<LOQ	
Fate Sees_202	1,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	51,2	<LOQ	<LOQ	4,3	<LOQ	<LOQ	<LOQ	75,2	<LOQ	<LOQ	<LOQ	
Fate Sees_203	0,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	15,3	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	14,6	<LOQ	<LOQ	<LOQ	
Fate Sees_204	0,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	74,5	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	35,9	<LOQ	<LOQ	<LOQ	
Fate Sees_205	0,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	67,5	<LOQ	<LOQ	3,0	<LOQ	<LOQ	<LOQ	27,2	<LOQ	<LOQ	<LOQ	
Fate Sees_208	7,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	29,9	<LOQ	<LOQ	1,6	<LOQ	<LOQ	<LOQ	38,7	<LOQ	<LOQ	7,1	
Fate Sees_209	0,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,3	<LOQ	<LOQ	1,0	<LOQ	<LOQ	<LOQ	93,7	<LOQ	1,8	<LOQ	
Fate Sees_210	3,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	10,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	32,4	<LOQ	<LOQ	1,7	
Fate Sees_211	2,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	2,4	<LOQ	<LOQ	4,6	<LOQ	<LOQ	1,5	18,2	<LOQ	2,5	<LOQ	
Fate Sees_214	5,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,3	<LOQ	<LOQ	3,0	<LOQ	<LOQ	<LOQ	36,0	<LOQ	<LOQ	<LOQ	
Fate Sees_226	2,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	7,8	<LOQ	<LOQ	1,9	<LOQ	<LOQ	<LOQ	8,3	<LOQ	<LOQ	<LOQ	
Fate Sees_227	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	6,3	<LOQ	<LOQ	<LOQ	
Fate Sees_228	0,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,4	<LOQ	<LOQ	0,9	<LOQ	<LOQ	10,2	<LOQ	<LOQ	1,3	
Fate Sees_229	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	
Fate Sees_233	3,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	9,7	<LOQ	<LOQ	1,5	<LOQ	<LOQ	<LOQ	18,2	<LOQ	<LOQ	<LOQ	
Fate Sees_235	5,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	0,5	<LOQ	<LOQ	<LOQ	15,7	<LOQ	<LOQ	<LOQ	
Fate Sees_236	2,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	24,5	<LOQ	<LOQ	1,0	<LOQ	<LOQ	<LOQ	9,4	<LOQ	<LOQ	<LOQ	
Fate Sees_239	1,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	20,9	<LOQ	<LOQ	1,1	<LOQ	<LOQ	<LOQ	21,0	<LOQ	<LOQ	<LOQ	
Fate Sees_242	2,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	10,1	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	12,9	<LOQ	<LOQ	<LOQ	
Fate Sees_243	3,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	<LOQ	<LOQ	1,9	<LOQ	<LOQ	<LOQ	46,0	<LOQ	1,6	<LOQ	
Fate Sees_245	1,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	22,5	<LOQ	<LOQ	3,5	<LOQ	<LOQ	<LOQ	16,8	<LOQ	<LOQ	<LOQ	
Fate Sees_247	2,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	13,1	<LOQ	<LOQ	0,6	<LOQ	<LOQ	<LOQ	14,1	<LOQ	<LOQ	1,2	
Fate Sees_248	3,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	0,9	<LOQ	<LOQ	<LOQ	39,1	<LOQ	<LOQ	<LOQ	
Fate Sees_250	1,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	151,7	<LOQ	<LOQ	0,4	<LOQ	<LOQ	<LOQ	20,9	<LOQ	<LOQ	2,5	
Fate Sees_252	4,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	178,0	<LOQ	<LOQ	1,5	<LOQ	<LOQ	<LOQ	61,1	<LOQ	<LOQ	<LOQ	
Fate Sees_253	0,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	86,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	18,6	<LOQ	0,5	<LOQ	
Fate Sees_254	9,4	33,0	<LOQ	<LOQ	<LOQ	<LOQ	0,4	143,0	<LOQ	4,7	<LOQ	<LOQ	2,2	47,8	<LOQ	0,7	6,6	
Fate Sees_256	0,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	43,5	<LOQ	<LOQ	0,5	<LOQ	<LOQ	<LOQ	39,5	<LOQ	<LOQ	2,8	
Fate Sees_257	1,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	15,1	<LOQ	<LOQ	2,6	<LOQ	<LOQ	<LOQ	16,0	<LOQ	<LOQ	<LOQ	
Fate Sees_258	1,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	24,9	<LOQ	<LOQ	1,4	<LOQ	<LOQ	<LOQ	21,1	<LOQ	<LOQ	<LOQ	
Fate Sees_259	8,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	108,2	<LOQ	2,9	<LOQ	<LOQ	1,7	16,9	<LOQ	1,0	<LOQ	
Fate Sees_260	7,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	25,0	<LOQ	<LOQ	1,8	<LOQ	<LOQ	<LOQ	57,0	<LOQ	<LOQ	<LOQ	
Fate Sees_261	4,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	19,5	<LOQ	<LOQ	0,8	<LOQ	<LOQ	<LOQ	60,9	<LOQ	<LOQ	5,5	
Fate Sees_262	4,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	28,4	<LOQ	<LOQ	1,5	<LOQ	<LOQ	<LOQ	31,8	<LOQ	<LOQ	<LOQ	
Fate Sees_263	4,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	6,5	<LOQ	<LOQ	1,0	<LOQ	<LOQ	<LOQ	56,0	<LOQ	<LOQ	<LOQ	
Fate Sees_264	4,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,3	8,7	<LOQ	1,1	<LOQ	<LOQ	<LOQ	29,7	<LOQ	<LOQ	<LOQ	
Fate Sees_265	13,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,1	105,5	<LOQ	0,3	<LOQ	<LOQ	<LOQ	133,5	<LOQ	<LOQ	<LOQ	
Fate Sees_266	3,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	20,6	<LOQ	0,2	<LOQ	<LOQ	<LOQ	79,8	<LOQ	<LOQ	<LOQ	
Fate Sees_267	8,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,4	19,0	<LOQ	1,0	<LOQ	<LOQ	<LOQ	43,6	<LOQ	0,6	<LOQ	
Fate Sees_268	12,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	23,5	<LOQ	0,8	<LOQ	<LOQ	<LOQ	81,0	<LOQ	1,3	43,7	

Annex 8: Results for pharmaceuticals; Lab: UMEÅ University, Sweden. (continued)

Sample	Codeine	Cyproheptadine	Dicycloverin	Diltiazem	Diphenhydramin	Duloxetine	Eprosartan	Etonogestrel	Fenofibrate	Fentanyl	Fexofenadine	Flecainide	Fluconazole	Fluoxetine	Flupetixol	Fluphenazine	Flutamid	Glibenclamide
LOQ [ng/l]	0,5	5	5	0,5	0,05	1	5	0,5	10	0,5	5	0,1	0,5	5	5	10	5	10
Fate Sees_136	2,4	<LOQ	<LOQ	<LOQ	10,5	<LOQ	16,2	<LOQ	<LOQ	<LOQ	66,6	5,0	161,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_137	43,9	<LOQ	<LOQ	3,6	9,5	<LOQ	207,3	<LOQ	<LOQ	<LOQ	121,8	9,2	227,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_138	1,3	<LOQ	<LOQ	<LOQ	1,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	552,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_139																		
Fate Sees_140	161,6	<LOQ	<LOQ	57,9	5,3	<LOQ	55,5	<LOQ	<LOQ	<LOQ	250,8	303,8	36,9	11,9	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_141																		
Fate Sees_142	137,2	<LOQ	<LOQ	22,8	4,1	<LOQ	83,0	<LOQ	<LOQ	<LOQ	293,8	66,1	107,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_143	11,4	<LOQ	<LOQ	1,2	2,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	2,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_144	5,6	<LOQ	<LOQ	<LOQ	22,5	<LOQ	29,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	5,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_145	20,9	<LOQ	<LOQ	6,8	9,0	<LOQ	11,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	120,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_146	14,2	<LOQ	<LOQ	8,0	2,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,7	89,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_147	5,8	<LOQ	<LOQ	5,1	4,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	23,0	181,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_148	2,9	<LOQ	<LOQ	5,0	0,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,8	46,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_149	48,6	<LOQ	<LOQ	7,2	7,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	52,6	4,9	188,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_150	7,6	<LOQ	<LOQ	0,7	0,5	6,3	22,3	<LOQ	16,9	<LOQ	11,9	2,4	20,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_151	7,7	<LOQ	<LOQ	<LOQ	0,9	<LOQ	<LOQ	<LOQ	14,0	<LOQ	<LOQ	0,7	7,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_152	188,9	<LOQ	<LOQ	<LOQ	1,3	<LOQ	<LOQ	<LOQ	14,0	<LOQ	14,5	3,7	7,5	<LOQ	<LOQ	<LOQ	<LOQ	28,0
Fate Sees_153	13,0	<LOQ	<LOQ	<LOQ	11,4	1,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,5	0,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_155	2,1	<LOQ	<LOQ	0,6	0,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	5,3	0,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_156	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_157	5,0	<LOQ	<LOQ	0,6	6,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	14,7	20,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_158	36,8	<LOQ	<LOQ	<LOQ	0,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	5,8	1,0	0,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_159	9,5	<LOQ	<LOQ	<LOQ	0,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,4	6,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_160	14,1	325,1	<LOQ	0,6	0,3	<LOQ	<LOQ	<LOQ	<LOQ	1,1	<LOQ	3,0	25,6	5,2	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_161	1,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,4	0,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_162	0,6	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,6	4,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_163	1,4	<LOQ	<LOQ	<LOQ	0,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1,4	0,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_164	1,6	<LOQ	<LOQ	<LOQ	4,3	1,8	<LOQ	<LOQ	<LOQ	<LOQ	26,2	31,6	203,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_165	12,3	<LOQ	<LOQ	1,4	2,6	<LOQ	32,7	<LOQ	<LOQ	<LOQ	32,9	27,9	379,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_166	11,9	<LOQ	<LOQ	13,8	12,1	<LOQ	12,5	<LOQ	<LOQ	<LOQ	68,2	113,4	412,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_167	6,1	<LOQ	<LOQ	22,5	16,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	173,1	398,8	413,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_168	6,0	18,3	<LOQ	7,1	5,6	<LOQ	28,0	<LOQ	<LOQ	0,6	18,8	36,1	198,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_169	21,6	<LOQ	<LOQ	6,1	5,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	402,4	1,1	237,0	18,5	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_170	826,4	<LOQ	<LOQ	20,1	17,4	<LOQ	6810,2	<LOQ	<LOQ	0,5	497,1	95,2	437,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_171	78,0	<LOQ	<LOQ	7,8	9,1	<LOQ	441,8	<LOQ	<LOQ	<LOQ	422,6	168,6	117,1	5,9	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_175	1,6	<LOQ	<LOQ	<LOQ	0,2	<LOQ	10,4	<LOQ	<LOQ	<LOQ	256,7	<LOQ	11,7	<LOQ	<LOQ	<LOQ	<LOQ	10,8
Fate Sees_177	0,8	<LOQ	<LOQ	<LOQ	1,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	353,0	0,6	67,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_179	651,8	<LOQ	<LOQ	19,4	8,2	<LOQ	4236,0	<LOQ	<LOQ	<LOQ	428,3	95,1	332,9	9,8	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_180	20,0	<LOQ	<LOQ	17,8	10,4	<LOQ	290,1	<LOQ	<LOQ	<LOQ	231,2	106,0	139,7	10,9	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_181	233,2	<LOQ	<LOQ	11,9	6,6	<LOQ	796,8	<LOQ	<LOQ	<LOQ	423,5	79,3	191,2	8,4	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_182	33,2	<LOQ	<LOQ	13,1	7,0	<LOQ	800,5	<LOQ	<LOQ	<LOQ	443,0	121,0	122,4	7,7	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_183	22,3	<LOQ	<LOQ	64,4	41,4	<LOQ	261,7	<LOQ	<LOQ	<LOQ	<LOQ	7,8	299,4	5,8	7,9	<LOQ	<LOQ	<LOQ
Fate Sees_184	<LOQ	<LOQ	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,8	283,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_189	3,3	<LOQ	<LOQ	<LOQ	9,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	129,0	0,2	0,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Fate Sees_190	0,7	<LOQ	<LOQ	<LOQ	6,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	40,3	0,9	30,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_191	33,6	<LOQ	<LOQ	<LOQ	10,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	80,9	3,8	19,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_193	4,6	<LOQ	<LOQ	<LOQ	1,3	2,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	34,5	10,8	18,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_194	45,9	<LOQ	<LOQ	<LOQ	6,0	25,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	129,8	13,1	65,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_195	310,0	<LOQ	<LOQ	<LOQ	8,2	22,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	191,9	10,1	97,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_196	312,3	<LOQ	<LOQ	<LOQ	12,0	15,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	140,6	11,1	69,8	5,9	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_198	6,0	<LOQ	<LOQ	<LOQ	2,5	16,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	15,4	8,5	12,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_199	15,0	<LOQ	<LOQ	<LOQ	5,8	16,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	18,9	6,3	20,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_202	11,3	<LOQ	<LOQ	<LOQ	8,2	91,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	35,8	7,4	64,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_203	5,0	<LOQ	<LOQ	<LOQ	1,9	4,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	19,2	5,0	87,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_204	18,3	<LOQ	<LOQ	<LOQ	6,0	18,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	21,4	5,3	50,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_205	25,0	<LOQ	<LOQ	<LOQ	2,2	39,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	31,9	5,4	17,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_208	85,1	<LOQ	<LOQ	<LOQ	12,5	20,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	64,9	100,7	103,9	7,2	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_209	70,2	<LOQ	<LOQ	<LOQ	9,0	44,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	48,9	74,4	111,8	8,7	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_210	46,1	<LOQ	<LOQ	<LOQ	42,0	6,6	<LOQ	<LOQ	<LOQ	<LOQ	0,8	<LOQ	0,6	13,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_211	199,3	<LOQ	<LOQ	<LOQ	4,9	3,5	<LOQ	<LOQ	<LOQ	<LOQ	11,9	<LOQ	78,7	14,5	99,4	5,7	<LOQ	<LOQ	5,8
Fate Sees_214	13,8	<LOQ	<LOQ	<LOQ	7,4	6,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	58,0	25,1	116,0	6,0	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_226	13,7	<LOQ	<LOQ	<LOQ	12,0	0,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	59,7	46,3	138,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_227	4,0	<LOQ	<LOQ	<LOQ	17,1	13,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	45,8	0,5	53,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_228	5,5	<LOQ	<LOQ	<LOQ	32,0	12,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	57,1	1,9	31,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_229	2,3	<LOQ	<LOQ	<LOQ	8,5	4,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	55,5	0,3	40,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_233	23,6	<LOQ	<LOQ	<LOQ	14,5	3,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	384,2	108,3	63,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_235	50,3	<LOQ	<LOQ	<LOQ	12,5	1,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	427,2	93,8	35,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_236	46,3	<LOQ	<LOQ	<LOQ	25,4	1,7	<LOQ	<LOQ	<LOQ	<LOQ	1,6	321,8	41,0	184,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_239	58,8	<LOQ	<LOQ	<LOQ	19,5	1,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	598,3	61,8	45,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_242	28,0	<LOQ	<LOQ	<LOQ	6,4	1,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	291,9	72,5	36,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_243	58,4	<LOQ	<LOQ	<LOQ	45,9	3,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	545,6	109,8	33,1	6,1	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_245	4,5	<LOQ	<LOQ	<LOQ	54,8	3,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	816,4	90,7	85,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_247	29,7	<LOQ	<LOQ	<LOQ	16,1	7,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	587,5	126,1	61,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_248	12,2	<LOQ	<LOQ	<LOQ	14,6	4,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	859,1	64,4	92,1	7,9	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_250	63,2	<LOQ	<LOQ	<LOQ	3,0	26,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	245,7	59,1	125,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_252	21,5	<LOQ	<LOQ	<LOQ	4,5	57,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	144,3	54,9	70,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_253	1,9	<LOQ	<LOQ	<LOQ	<LOQ	141,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1287,1	8,7	2,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_254	98,1	<LOQ	<LOQ	<LOQ	32,5	41,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	59,5	28,4	344,1	21,5	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_256	2,6	<LOQ	<LOQ	<LOQ	0,6	12,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	128,5	47,0	38,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_257	55,5	<LOQ	<LOQ	<LOQ	25,5	0,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	421,5	127,0	104,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_258	70,1	<LOQ	<LOQ	<LOQ	26,5	2,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	484,9	107,7	138,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_259	43,4	<LOQ	<LOQ	<LOQ	12,8	37,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	211,8	29,3	598,1	16,6	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_260	143,0	<LOQ	<LOQ	<LOQ	10,7	3,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	39,5	13,3	268,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_261	134,6	<LOQ	<LOQ	<LOQ	7,0	2,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	58,8	9,2	92,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_262	52,1	<LOQ	<LOQ	<LOQ	2,0	4,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	27,6	14,7	77,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_263	79,0	<LOQ	<LOQ	<LOQ	5,4	2,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	40,4	9,8	48,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_264	150,0	<LOQ	<LOQ	<LOQ	12,5	0,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	66,9	15,4	39,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_265	684,6	<LOQ	<LOQ	<LOQ	9,2	26,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	213,2	61,9	420,6	7,6	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_266	159,1	<LOQ	<LOQ	<LOQ	6,1	1,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	86,6	5,9	46,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_267	102,3	<LOQ	<LOQ	<LOQ	16,2	2,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	25,9	49,8	74,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_268	208,5	<LOQ	<LOQ	<LOQ	40,7	3,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	58,2	21,4	135,3	6,9	<LOQ	<LOQ	<LOQ	<LOQ

Annex 8: Results for pharmaceuticals; Lab: UMEÅ University, Sweden. (continued)

Sample	Glimepiride	Haloperidol	Hydroxyzine	Ibuprofen	Loperamide	Maprotilin	Meclozine	Memantin	Mianserin	Miconazole	Nefazodon	Orphenadrin	Oxazepam	Paroxetin	Perphenazine	Pizotifen	Promethazin	Ranitidine
LOQ [ng/l]	10	0,1	0,5	0,5	0,5	5	5	0,5	1	5	0,5	0,1	5	10	10	0,5	10	5
Fate Sees_136	<LOQ	<LOQ	0,8	6,8	<LOQ	<LOQ	<LOQ	4,1	<LOQ	<LOQ	<LOQ	3,6	21,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_137	<LOQ	0,3	<LOQ	55,3	<LOQ	<LOQ	<LOQ	6,0	<LOQ	<LOQ	<LOQ	1,4	16,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_138	<LOQ	<LOQ	<LOQ	663,7	<LOQ	<LOQ	<LOQ	0,9	5,4	<LOQ	<LOQ	<LOQ	1765,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_139	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_140	<LOQ	0,2	0,6	2184,8	<LOQ	<LOQ	<LOQ	15,4	1,2	<LOQ	<LOQ	0,1	427,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_141	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_142	<LOQ	<LOQ	2,0	1262,5	<LOQ	<LOQ	<LOQ	15,2	1,3	<LOQ	<LOQ	0,3	467,6	<LOQ	<LOQ	<LOQ	<LOQ	7,8
Fate Sees_143	<LOQ	0,2	<LOQ	2,2	<LOQ	<LOQ	<LOQ	12,8	<LOQ	<LOQ	<LOQ	0,4	44,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_144	<LOQ	<LOQ	<LOQ	6,6	<LOQ	<LOQ	<LOQ	12,8	<LOQ	<LOQ	<LOQ	0,9	42,1	<LOQ	<LOQ	<LOQ	<LOQ	6,1
Fate Sees_145	<LOQ	<LOQ	<LOQ	1,8	<LOQ	<LOQ	<LOQ	7,2	<LOQ	<LOQ	<LOQ	0,8	33,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_146	<LOQ	0,1	0,6	4,8	<LOQ	<LOQ	<LOQ	2,5	<LOQ	<LOQ	<LOQ	1,0	64,7	<LOQ	<LOQ	<LOQ	<LOQ	10,7
Fate Sees_147	<LOQ	0,4	<LOQ	20,4	<LOQ	<LOQ	<LOQ	3,7	<LOQ	<LOQ	<LOQ	0,7	72,3	<LOQ	<LOQ	<LOQ	<LOQ	19,4
Fate Sees_148	<LOQ	0,3	<LOQ	2,2	<LOQ	<LOQ	<LOQ	28,7	<LOQ	<LOQ	<LOQ	0,5	25,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_149	<LOQ	0,1	1,1	16,4	<LOQ	<LOQ	<LOQ	4,0	<LOQ	<LOQ	<LOQ	1,3	47,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_150	<LOQ	0,7	5,3	7,3	<LOQ	9,1	<LOQ	1312,1	5,8	<LOQ	<LOQ	0,9	34,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_151	<LOQ	0,3	2,5	4,2	<LOQ	<LOQ	<LOQ	1,7	3,8	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_152	<LOQ	2,1	9,6	7,9	<LOQ	16,5	<LOQ	40,0	1,1	<LOQ	<LOQ	2,4	13,8	<LOQ	<LOQ	<LOQ	<LOQ	18,6
Fate Sees_153	<LOQ	0,4	<LOQ	3,9	<LOQ	<LOQ	<LOQ	0,6	<LOQ	14,8	0,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_155	<LOQ	0,8	<LOQ	4,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_156	<LOQ	<LOQ	<LOQ	3,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_157	<LOQ	0,1	<LOQ	13,3	<LOQ	<LOQ	<LOQ	0,9	5,2	<LOQ	<LOQ	0,2	20,3	<LOQ	<LOQ	<LOQ	<LOQ	17,0
Fate Sees_158	<LOQ	30,8	0,7	17884,0	24,3	<LOQ	<LOQ	<LOQ	1,3	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_159	<LOQ	0,5	<LOQ	3,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,8	5,4	<LOQ	<LOQ	<LOQ	<LOQ	32,2
Fate Sees_160	<LOQ	2691,5	<LOQ	5,9	2409,1	<LOQ	<LOQ	0,6	62,3	<LOQ	<LOQ	0,1	62,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_161	<LOQ	<LOQ	<LOQ	2,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_162	<LOQ	0,1	<LOQ	4,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_163	<LOQ	0,1	<LOQ	7,0	<LOQ	<LOQ	<LOQ	0,9	<LOQ	<LOQ	<LOQ	0,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_164	<LOQ	1,3	0,5	57,4	<LOQ	<LOQ	<LOQ	1,5	<LOQ	<LOQ	<LOQ	0,5	58,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_165	<LOQ	0,2	2,3	318,9	<LOQ	<LOQ	<LOQ	5,3	<LOQ	<LOQ	<LOQ	0,2	55,1	<LOQ	<LOQ	<LOQ	<LOQ	20,9
Fate Sees_166	<LOQ	0,1	0,6	192,1	<LOQ	<LOQ	<LOQ	4,6	<LOQ	<LOQ	<LOQ	0,6	62,4	<LOQ	<LOQ	<LOQ	<LOQ	29,6
Fate Sees_167	<LOQ	0,2	<LOQ	704,0	<LOQ	<LOQ	<LOQ	5,0	<LOQ	<LOQ	<LOQ	1,1	69,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_168	<LOQ	113,5	<LOQ	266,1	169,2	<LOQ	<LOQ	1,2	11,0	<LOQ	<LOQ	0,4	29,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_169	<LOQ	0,7	<LOQ	287,7	<LOQ	<LOQ	<LOQ	10,9	<LOQ	<LOQ	<LOQ	0,5	43,9	<LOQ	<LOQ	<LOQ	<LOQ	11,4
Fate Sees_170	<LOQ	1,7	2,2	6,0	<LOQ	<LOQ	<LOQ	45,8	1,5	<LOQ	<LOQ	27,0	280,3	<LOQ	<LOQ	<LOQ	<LOQ	9,2
Fate Sees_171	<LOQ	0,5	1,2	1,7	<LOQ	<LOQ	<LOQ	43,4	1,7	<LOQ	<LOQ	37,0	242,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_175	<LOQ	0,7	<LOQ	80,9	<LOQ	<LOQ	<LOQ	2,7	<LOQ	<LOQ	<LOQ	<LOQ	26,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_177	<LOQ	<LOQ	<LOQ	121,7	<LOQ	<LOQ	<LOQ	2,4	<LOQ	<LOQ	<LOQ	<LOQ	37,6	<LOQ	<LOQ	<LOQ	<LOQ	5,6
Fate Sees_179	<LOQ	0,1	2,4	1,7	<LOQ	<LOQ	<LOQ	12,8	1,5	<LOQ	<LOQ	45,8	529,5	<LOQ	<LOQ	<LOQ	<LOQ	10,2
Fate Sees_180	<LOQ	0,4	1,5	2,7	<LOQ	<LOQ	<LOQ	18,9	4,5	<LOQ	<LOQ	46,7	368,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_181	<LOQ	0,2	2,4	12,4	<LOQ	<LOQ	<LOQ	20,4	1,5	<LOQ	<LOQ	39,0	471,3	<LOQ	<LOQ	<LOQ	<LOQ	6,4
Fate Sees_182	<LOQ	0,9	0,9	5,2	<LOQ	<LOQ	<LOQ	22,2	<LOQ	<LOQ	<LOQ	23,5	378,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_183	<LOQ	2,2	2,2	916,4	<LOQ	<LOQ	<LOQ	10,0	<LOQ	<LOQ	<LOQ	38,9	20,2	<LOQ	<LOQ	<LOQ	<LOQ	43,6
Fate Sees_184	<LOQ	<LOQ	<LOQ	265,4	<LOQ	<LOQ	<LOQ	2,2	<LOQ	<LOQ	<LOQ	1,1	7,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_189	<LOQ	0,1	<LOQ	334,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,5	95,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

Fate Sees_190	<LOQ	<LOQ	<LOQ	50,9	<LOQ	<LOQ	<LOQ	1,6	<LOQ	<LOQ	<LOQ	<LOQ	0,2	77,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_191	<LOQ	0,1	<LOQ	179,9	<LOQ	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	<LOQ	0,6	14,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_193	<LOQ	<LOQ	<LOQ	129,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,2	50,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_194	<LOQ	0,2	1,2	221,8	0,5	14,3	<LOQ	2,3	<LOQ	<LOQ	<LOQ	<LOQ	0,2	179,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_195	<LOQ	0,1	<LOQ	231,6	<LOQ	<LOQ	<LOQ	6,7	<LOQ	<LOQ	<LOQ	<LOQ	1,0	64,3	<LOQ	<LOQ	<LOQ	<LOQ	18,0
Fate Sees_196	<LOQ	0,8	<LOQ	49,5	<LOQ	<LOQ	<LOQ	4,3	<LOQ	<LOQ	<LOQ	<LOQ	0,2	17,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_198	<LOQ	0,2	2,8	16,1	<LOQ	<LOQ	<LOQ	1,7	<LOQ	<LOQ	<LOQ	<LOQ	0,8	77,7	<LOQ	<LOQ	<LOQ	<LOQ	5,1
Fate Sees_199	<LOQ	0,6	<LOQ	17,9	<LOQ	<LOQ	<LOQ	3,8	<LOQ	<LOQ	<LOQ	<LOQ	0,5	53,6	<LOQ	<LOQ	<LOQ	<LOQ	6,8
Fate Sees_202	<LOQ	0,5	<LOQ	14,3	<LOQ	<LOQ	<LOQ	11,9	<LOQ	<LOQ	<LOQ	<LOQ	0,8	219,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_203	<LOQ	0,1	<LOQ	4,1	<LOQ	<LOQ	<LOQ	3,6	<LOQ	<LOQ	<LOQ	<LOQ	0,9	98,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_204	<LOQ	<LOQ	0,6	9,0	<LOQ	<LOQ	<LOQ	3,3	<LOQ	<LOQ	<LOQ	<LOQ	1,0	206,5	<LOQ	<LOQ	<LOQ	<LOQ	8,7
Fate Sees_205	<LOQ	0,2	0,9	32,2	<LOQ	<LOQ	<LOQ	7,0	<LOQ	<LOQ	<LOQ	<LOQ	1,8	190,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_208	<LOQ	0,3	<LOQ	291,7	<LOQ	<LOQ	<LOQ	22,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	61,3	<LOQ	<LOQ	<LOQ	<LOQ	9,7
Fate Sees_209	<LOQ	<LOQ	2,9	1203,2	<LOQ	<LOQ	<LOQ	19,3	<LOQ	<LOQ	<LOQ	0,6	0,1	51,1	<LOQ	<LOQ	0,7	<LOQ	<LOQ
Fate Sees_210	<LOQ	0,2	<LOQ	423,6	<LOQ	<LOQ	<LOQ	23,1	1,7	<LOQ	<LOQ	<LOQ	<LOQ	60,9	<LOQ	<LOQ	<LOQ	<LOQ	21,1
Fate Sees_211	<LOQ	1,3	5,5	247,0	2,2	<LOQ	<LOQ	7,1	<LOQ	<LOQ	<LOQ	<LOQ	10,4	229,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_214	<LOQ	0,2	1,3	221,6	<LOQ	<LOQ	<LOQ	3,4	<LOQ	<LOQ	<LOQ	<LOQ	6,1	224,6	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_226	<LOQ	1,2	<LOQ	393,5	<LOQ	<LOQ	<LOQ	2,9	<LOQ	<LOQ	<LOQ	<LOQ	1,5	18,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_227	<LOQ	0,3	<LOQ	10,2	<LOQ	<LOQ	<LOQ	5,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	25,2	<LOQ	<LOQ	<LOQ	<LOQ	14,6
Fate Sees_228	<LOQ	0,5	<LOQ	23,8	<LOQ	<LOQ	<LOQ	3,7	<LOQ	<LOQ	<LOQ	<LOQ	0,2	60,8	<LOQ	<LOQ	<LOQ	<LOQ	30,8
Fate Sees_229	<LOQ	0,2	<LOQ	8,0	<LOQ	<LOQ	<LOQ	1,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	62,2	<LOQ	<LOQ	<LOQ	<LOQ	25,2
Fate Sees_233	<LOQ	1,1	<LOQ	817,8	2,2	<LOQ	<LOQ	0,9	<LOQ	<LOQ	<LOQ	<LOQ	0,9	433,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_235	<LOQ	0,4	2,0	640,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,8	27,8	<LOQ	<LOQ	<LOQ	<LOQ	8,5
Fate Sees_236	<LOQ	0,3	3,8	405,6	<LOQ	<LOQ	<LOQ	1,4	<LOQ	<LOQ	<LOQ	<LOQ	0,4	198,2	<LOQ	<LOQ	<LOQ	<LOQ	16,7
Fate Sees_239	<LOQ	0,1	<LOQ	859,5	<LOQ	<LOQ	<LOQ	1,1	1,2	<LOQ	<LOQ	<LOQ	0,6	287,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_242	<LOQ	0,3	<LOQ	493,4	<LOQ	<LOQ	<LOQ	0,8	<LOQ	<LOQ	<LOQ	<LOQ	0,6	468,1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_243	<LOQ	0,6	2,1	567,4	<LOQ	<LOQ	<LOQ	1,6	<LOQ	<LOQ	<LOQ	<LOQ	0,2	565,0	<LOQ	<LOQ	<LOQ	<LOQ	9,7
Fate Sees_245	<LOQ	<LOQ	<LOQ	1145,6	<LOQ	<LOQ	<LOQ	1,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	325,3	<LOQ	<LOQ	<LOQ	<LOQ	11,3
Fate Sees_247	<LOQ	0,2	1,4	861,1	<LOQ	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	<LOQ	0,5	256,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_248	<LOQ	0,2	2,9	483,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,8	621,2	<LOQ	<LOQ	<LOQ	<LOQ	23,0
Fate Sees_250	<LOQ	0,5	0,8	341,3	<LOQ	<LOQ	<LOQ	4,4	<LOQ	<LOQ	<LOQ	<LOQ	0,4	74,4	<LOQ	<LOQ	<LOQ	<LOQ	21,0
Fate Sees_252	<LOQ	1,2	<LOQ	236,8	1,5	<LOQ	<LOQ	10,5	<LOQ	<LOQ	<LOQ	<LOQ	0,3	49,0	<LOQ	<LOQ	<LOQ	<LOQ	6,5
Fate Sees_253	<LOQ	0,4	0,6	3,4	<LOQ	<LOQ	<LOQ	0,9	<LOQ	<LOQ	<LOQ	<LOQ	0,6	<LOQ	<LOQ	<LOQ	0,7	<LOQ	<LOQ
Fate Sees_254	<LOQ	1,0	3,3	1372,3	<LOQ	<LOQ	<LOQ	33,6	4,6	<LOQ	<LOQ	<LOQ	0,9	149,9	<LOQ	<LOQ	<LOQ	<LOQ	37,9
Fate Sees_256	<LOQ	<LOQ	<LOQ	653,1	<LOQ	<LOQ	<LOQ	10,9	<LOQ	<LOQ	<LOQ	<LOQ	0,2	51,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_257	<LOQ	0,2	<LOQ	799,8	<LOQ	<LOQ	<LOQ	0,7	<LOQ	<LOQ	<LOQ	<LOQ	0,2	484,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_258	<LOQ	0,4	1,2	588,8	<LOQ	<LOQ	<LOQ	<LOQ	1,0	<LOQ	<LOQ	<LOQ	1,0	388,0	<LOQ	<LOQ	<LOQ	<LOQ	9,7
Fate Sees_259	<LOQ	0,3	0,7	1259,0	<LOQ	<LOQ	<LOQ	50,6	1,5	<LOQ	<LOQ	<LOQ	0,3	224,0	<LOQ	<LOQ	<LOQ	<LOQ	8,1
Fate Sees_260	<LOQ	0,5	0,9	88,2	<LOQ	<LOQ	<LOQ	6,8	4,7	<LOQ	<LOQ	<LOQ	3,3	121,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_261	<LOQ	0,5	2,5	85,4	<LOQ	<LOQ	<LOQ	3,6	1,4	<LOQ	<LOQ	<LOQ	4,7	192,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_262	<LOQ	0,2	1,4	237,4	<LOQ	<LOQ	<LOQ	3,6	1,4	<LOQ	<LOQ	<LOQ	4,3	162,2	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_263	<LOQ	0,3	0,9	45,4	<LOQ	<LOQ	<LOQ	5,2	<LOQ	<LOQ	<LOQ	<LOQ	1,6	89,7	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_264	<LOQ	0,4	3,7	239,5	<LOQ	<LOQ	<LOQ	3,0	<LOQ	<LOQ	<LOQ	<LOQ	6,6	182,7	<LOQ	<LOQ	<LOQ	<LOQ	5,5
Fate Sees_265	<LOQ	0,4	3,3	294,0	<LOQ	<LOQ	<LOQ	9,5	<LOQ	<LOQ	<LOQ	<LOQ	3,3	157,9	<LOQ	<LOQ	<LOQ	<LOQ	24,7
Fate Sees_266	<LOQ	0,3	6,1	65,7	<LOQ	<LOQ	<LOQ	25,6	3,4	<LOQ	<LOQ	<LOQ	0,4	348,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_267	<LOQ	0,2	2,1	71,5	<LOQ	<LOQ	<LOQ	10,7	<LOQ	<LOQ	<LOQ	<LOQ	4,6	157,5	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_268	<LOQ	0,4	5,0	483,1	<LOQ	<LOQ	<LOQ	23,4	3,9	<LOQ	<LOQ	<LOQ	7,3	391,5	<LOQ	<LOQ	<LOQ	<LOQ	36,7

Annex 8: Results for pharmaceuticals; Lab: UMEÅ University, Sweden. (continued)

Sample	Repaglinide	Risperidone	Rosuvastatin	Sertraline	Tamoxifen	Telmisartan	Terbutalin	Tramadol	Trihexyphenidyl	Venlafaxin	Verapamil	Zolpidem
LOQ [ng/l]	0,5	0,1	10	10	5	50	0,5	0,5	0,1	0,5	10	0,5
Fate Sees_136	1,7	1,4	<LOQ	<LOQ	<LOQ	3114,0	0,6	25,0	<LOQ	69,0	<LOQ	<LOQ
Fate Sees_137	2,3	2,1	25,6	<LOQ	<LOQ	3426,0	0,9	98,3	0,1	51,4	<LOQ	<LOQ
Fate Sees_138	2,0	1,8	<LOQ	<LOQ	<LOQ	59,0	<LOQ	1165,7	<LOQ	276,5	<LOQ	0,7
Fate Sees_139												
Fate Sees_140	6,6	2,2	<LOQ	<LOQ	<LOQ	342,9	<LOQ	681,6	0,3	205,2	<LOQ	0,9
Fate Sees_141												
Fate Sees_142	12,3	6,8	<LOQ	<LOQ	<LOQ	64,6	0,6	704,9	0,7	107,6	<LOQ	<LOQ
Fate Sees_143	2,0	1,8	<LOQ	<LOQ	<LOQ	1010,5	1,5	181,0	<LOQ	54,0	<LOQ	1,7
Fate Sees_144	1,6	3,4	<LOQ	<LOQ	<LOQ	1696,1	<LOQ	282,2	<LOQ	104,3	<LOQ	10,0
Fate Sees_145	<LOQ	5,5	<LOQ	<LOQ	<LOQ	1229,2	<LOQ	599,5	<LOQ	149,6	<LOQ	1,4
Fate Sees_146	1,5	29,5	<LOQ	<LOQ	<LOQ	274,3	<LOQ	593,9	0,2	44,6	<LOQ	1,8
Fate Sees_147	2,1	1,4	<LOQ	<LOQ	<LOQ	764,9	1,2	438,1	<LOQ	70,7	<LOQ	2,2
Fate Sees_148	0,7	4,3	<LOQ	<LOQ	<LOQ	665,7	0,8	308,0	<LOQ	134,7	<LOQ	18,3
Fate Sees_149	1,4	2,8	<LOQ	<LOQ	<LOQ	518,1	1,2	501,9	<LOQ	131,1	<LOQ	6,2
Fate Sees_150	11,1	9,6	<LOQ	15,9	<LOQ	<LOQ	3,9	19,7	<LOQ	54,2	<LOQ	0,5
Fate Sees_151	3,9	18,9	<LOQ	<LOQ	<LOQ	<LOQ	4,7	5,5	1,1	27,2	<LOQ	<LOQ
Fate Sees_152	9,8	6,4	<LOQ	26,1	9,8	<LOQ	3,1	6,9	1,7	21,0	<LOQ	<LOQ
Fate Sees_153	5,3	1,0	<LOQ	37,5	12,6	<LOQ	2,9	2,3	<LOQ	17,8	<LOQ	<LOQ
Fate Sees_155	3,7	1,6	<LOQ	<LOQ	<LOQ	<LOQ	2,1	2,6	0,2	52,9	<LOQ	<LOQ
Fate Sees_156	2,5	4,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1,7	<LOQ	<LOQ	<LOQ	<LOQ
Fate Sees_157	4,7	4,0	<LOQ	<LOQ	<LOQ	<LOQ	4,6	22,3	0,3	16,6	<LOQ	0,9
Fate Sees_158	1,8	2,2	978,9	18,3	<LOQ	<LOQ	1,2	0,7	0,2	11,4	<LOQ	0,6
Fate Sees_159	8,6	2,3	<LOQ	18,6	<LOQ	<LOQ	<LOQ	86,0	<LOQ	10,1	<LOQ	<LOQ
Fate Sees_160	4,4	4,8	<LOQ	<LOQ	<LOQ	<LOQ	1,0	3,0	0,2	16,3	<LOQ	2,8
Fate Sees_161	3,4	2,4	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0,5	<LOQ	2,9	<LOQ	<LOQ
Fate Sees_162	8,4	1,8	<LOQ	<LOQ	<LOQ	<LOQ	1,3	1,7	<LOQ	5,4	<LOQ	<LOQ
Fate Sees_163	7,7	3,4	<LOQ	<LOQ	<LOQ	<LOQ	2,2	43,9	0,3	6,9	<LOQ	<LOQ
Fate Sees_164	7,6	28,2	19,6	<LOQ	<LOQ	113,3	2,6	170,9	0,3	64,3	<LOQ	0,9
Fate Sees_165	9,0	8,8	<LOQ	15,5	<LOQ	283,2	0,7	250,7	0,9	123,1	<LOQ	0,9
Fate Sees_166	4,0	4,3	<LOQ	<LOQ	<LOQ	73,0	<LOQ	432,9	0,1	150,3	<LOQ	1,5
Fate Sees_167	4,0	2,1	<LOQ	<LOQ	<LOQ	309,2	0,8	1160,0	<LOQ	374,5	<LOQ	2,5
Fate Sees_168	1,5	2,6	<LOQ	<LOQ	<LOQ	169,6	<LOQ	169,0	<LOQ	77,6	<LOQ	1,3
Fate Sees_169	3,3	2,7	134,4	<LOQ	<LOQ	452,1	2,0	402,2	<LOQ	198,7	<LOQ	42,6
Fate Sees_170	2,2	3,7	175,6	<LOQ	<LOQ	1936,7	2,8	278,4	0,4	548,3	<LOQ	0,7
Fate Sees_171	2,0	15,2	81,4	<LOQ	<LOQ	662,3	1,6	251,7	<LOQ	125,3	<LOQ	0,6
Fate Sees_175	6,8	2,9	17,5	<LOQ	<LOQ	<LOQ	<LOQ	242,9	<LOQ	8,3	<LOQ	<LOQ
Fate Sees_177	5,4	2,3	<LOQ	<LOQ	<LOQ	<LOQ	0,5	186,9	<LOQ	31,2	<LOQ	<LOQ
Fate Sees_179	0,9	1,9	110,6	<LOQ	<LOQ	600,7	1,4	507,2	<LOQ	302,8	<LOQ	1,2
Fate Sees_180	1,4	3,4	23,1	<LOQ	<LOQ	248,3	1,2	287,7	0,4	271,3	<LOQ	0,7
Fate Sees_181	2,3	2,1	41,2	<LOQ	<LOQ	540,0	1,4	214,7	<LOQ	186,0	<LOQ	0,8
Fate Sees_182	1,7	2,7	43,0	<LOQ	<LOQ	375,3	2,2	213,3	0,1	233,9	<LOQ	1,3
Fate Sees_183	1,1	85,8	<LOQ	<LOQ	<LOQ	378,3	<LOQ	18,4	0,1	65,4	<LOQ	1,6
Fate Sees_184	1,2	10,2	<LOQ	<LOQ	<LOQ	66,6	1,3	1,5	0,2	1,4	<LOQ	<LOQ
Fate Sees_189	1,1	6,1	<LOQ	<LOQ	<LOQ	307,0	0,6	61,5	<LOQ	66,7	<LOQ	<LOQ

Fate Sees_190	1,2	2,0	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	127,7	<LOQ	57,8	<LOQ	<LOQ
Fate Sees_191	0,9	6,8	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	53,6	<LOQ	52,0	<LOQ	1,3
Fate Sees_193	1,3	1,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	61,9	<LOQ	71,7	<LOQ	<LOQ
Fate Sees_194	1,1	5,0	18,2	<LOQ	<LOQ	115,3	0,7	179,7	<LOQ	171,4	<LOQ	1,2
Fate Sees_195	2,5	1,7	36,4	<LOQ	<LOQ	131,6	0,9	228,6	0,2	133,7	<LOQ	<LOQ
Fate Sees_196	1,4	1,8	11,3	<LOQ	<LOQ	114,6	0,8	166,1	<LOQ	128,6	<LOQ	0,8
Fate Sees_198	1,5	43,1	11,3	<LOQ	<LOQ	83,8	0,9	137,3	0,1	61,1	<LOQ	0,9
Fate Sees_199	1,2	36,3	<LOQ	<LOQ	<LOQ	106,2	1,5	133,5	0,2	97,0	<LOQ	1,7
Fate Sees_202	1,9	2,5	<LOQ	<LOQ	<LOQ	87,0	2,1	211,5	<LOQ	169,7	<LOQ	1,7
Fate Sees_203	<LOQ	5,9	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	126,5	<LOQ	31,4	<LOQ	<LOQ
Fate Sees_204	2,4	3,7	<LOQ	<LOQ	<LOQ	155,6	0,7	244,5	0,1	77,2	<LOQ	0,9
Fate Sees_205	3,5	3,2	10,3	<LOQ	<LOQ	248,9	0,7	410,2	<LOQ	146,5	<LOQ	0,7
Fate Sees_208	1,2	4,3	<LOQ	<LOQ	<LOQ	531,9	1,0	257,1	<LOQ	168,0	<LOQ	1,1
Fate Sees_209	3,3	3,9	<LOQ	10,9	<LOQ	4344,8	3,0	398,5	0,8	184,1	<LOQ	<LOQ
Fate Sees_210	3,1	2,0	<LOQ	<LOQ	<LOQ	1058,0	0,6	222,7	<LOQ	149,0	<LOQ	1,4
Fate Sees_211	5,9	9,9	17,2	<LOQ	<LOQ	<LOQ	<LOQ	361,8	<LOQ	132,7	<LOQ	0,9
Fate Sees_214	2,1	3,3	<LOQ	<LOQ	<LOQ	<LOQ	1,0	309,7	0,3	102,5	<LOQ	1,6
Fate Sees_226	1,6	2,5	15,8	<LOQ	<LOQ	418,0	0,7	31,7	<LOQ	56,8	<LOQ	<LOQ
Fate Sees_227	0,7	2,5	<LOQ	<LOQ	<LOQ	<LOQ	0,8	55,9	0,5	22,9	<LOQ	<LOQ
Fate Sees_228	1,9	3,3	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	60,5	0,8	30,3	<LOQ	0,5
Fate Sees_229	2,0	2,1	<LOQ	<LOQ	<LOQ	<LOQ	1,1	41,0	0,3	18,1	<LOQ	<LOQ
Fate Sees_233	1,7	2,3	<LOQ	<LOQ	<LOQ	258,3	0,7	235,9	0,1	189,5	<LOQ	<LOQ
Fate Sees_235	1,4	23,4	33,0	<LOQ	<LOQ	230,5	0,7	249,6	<LOQ	125,1	<LOQ	<LOQ
Fate Sees_236	1,8	5,7	33,1	<LOQ	<LOQ	148,1	0,9	199,3	<LOQ	67,6	<LOQ	0,7
Fate Sees_239	1,6	2,6	<LOQ	<LOQ	<LOQ	153,3	<LOQ	186,4	<LOQ	156,2	<LOQ	<LOQ
Fate Sees_242	1,6	20,4	19,7	<LOQ	<LOQ	323,8	0,6	211,2	<LOQ	88,7	<LOQ	0,5
Fate Sees_243	1,6	11,9	<LOQ	<LOQ	<LOQ	288,3	1,2	252,5	0,2	121,8	<LOQ	<LOQ
Fate Sees_245	3,1	5,3	11,4	<LOQ	<LOQ	232,3	1,0	305,5	0,2	206,2	<LOQ	<LOQ
Fate Sees_247	1,2	12,3	11,5	<LOQ	<LOQ	73,5	0,7	218,4	<LOQ	71,5	<LOQ	<LOQ
Fate Sees_248	1,8	5,4	<LOQ	<LOQ	<LOQ	120,1	1,6	380,4	<LOQ	153,1	<LOQ	<LOQ
Fate Sees_250	2,3	17,1	<LOQ	<LOQ	<LOQ	413,0	1,8	456,3	0,5	70,6	<LOQ	<LOQ
Fate Sees_252	3,1	2,4	<LOQ	<LOQ	<LOQ	250,2	0,6	196,3	<LOQ	113,9	<LOQ	0,5
Fate Sees_253	5,7	3,3	<LOQ	<LOQ	<LOQ	417,7	2,7	0,8	0,3	65,8	<LOQ	<LOQ
Fate Sees_254	2,5	2,4	432,2	<LOQ	<LOQ	927,4	1,7	271,1	0,5	329,6	<LOQ	0,8
Fate Sees_256	1,5	3,7	<LOQ	<LOQ	<LOQ	313,6	<LOQ	84,6	0,1	72,5	<LOQ	<LOQ
Fate Sees_257	2,0	2,7	99,9	<LOQ	<LOQ	333,7	<LOQ	183,1	0,1	256,3	<LOQ	<LOQ
Fate Sees_258	2,1	3,2	<LOQ	<LOQ	<LOQ	267,9	1,0	221,9	<LOQ	228,9	<LOQ	<LOQ
Fate Sees_259	2,9	3,9	217,9	<LOQ	<LOQ	878,8	0,9	443,6	0,4	275,1	<LOQ	0,5
Fate Sees_260	3,4	3,4	39,2	<LOQ	<LOQ	<LOQ	0,8	502,6	0,3	118,2	<LOQ	1,4
Fate Sees_261	1,9	2,5	23,1	<LOQ	<LOQ	<LOQ	0,9	487,3	<LOQ	177,4	<LOQ	1,1
Fate Sees_262	3,5	2,5	<LOQ	13,3	<LOQ	<LOQ	0,8	358,5	0,1	138,2	<LOQ	0,9
Fate Sees_263	1,5	3,4	<LOQ	<LOQ	<LOQ	<LOQ	1,4	318,1	0,3	92,5	<LOQ	<LOQ
Fate Sees_264	3,3	7,6	15,3	<LOQ	<LOQ	<LOQ	0,8	307,9	<LOQ	87,1	<LOQ	0,5
Fate Sees_265	4,4	2,6	17,8	12,7	<LOQ	<LOQ	3,0	471,3	<LOQ	386,4	<LOQ	2,5
Fate Sees_266	4,2	2,4	<LOQ	11,6	<LOQ	<LOQ	2,4	834,2	0,2	239,1	<LOQ	1,6
Fate Sees_267	6,0	4,3	22,8	<LOQ	<LOQ	<LOQ	0,9	346,1	0,2	88,6	<LOQ	1,0
Fate Sees_268	3,4	3,1	14,2	10,9	<LOQ	<LOQ	1,6	595,8	<LOQ	129,2	<LOQ	1,6

Annex 9: Estrogenicity and dioxin-like toxicity by in vitro reporter gene bioassays; Lab: RECETOX, Masaryk University, Brno, Czech Republic.

“n.a.” = not analyzed.

Sample	Estrogenicity (EEQ)	Dioxin-like toxicity (TEQbio)
LOQ [ng/l]	0.5	0.1
Fate Sees_136	3,56	0,12
Fate Sees_137	<0,5	0,11
Fate Sees_138		
Fate Sees_139		
Fate Sees_140		
Fate Sees_141		
Fate Sees_142		
Fate Sees_143	<0,5	<0,1
Fate Sees_144	1,95	<0,1
Fate Sees_145	1,2	0,20
Fate Sees_146	<0,5	0,24
Fate Sees_147	1,26	0,18
Fate Sees_148	1,70	0,12
Fate Sees_149	2,12	0,16
Fate Sees_150	1,8	n.a.
Fate Sees_151	<0,5	n.a.
Fate Sees_152	<0,5	n.a.
Fate Sees_153	<0,5	n.a.
Fate Sees_155	<0,5	n.a.
Fate Sees_156	0,8	n.a.
Fate Sees_157	<0,5	n.a.
Fate Sees_158	<0,5	n.a.
Fate Sees_159	1,2	n.a.
Fate Sees_160	0,6	n.a.
Fate Sees_161	<0,5	n.a.
Fate Sees_162	<0,5	n.a.
Fate Sees_163	3,40	n.a.
Fate Sees_164	<0,5	n.a.
Fate Sees_165	<0,5	n.a.
Fate Sees_166	<0,5	n.a.
Fate Sees_167	<0,5	n.a.
Fate Sees_168	<0,5	n.a.
Fate Sees_169	4,15	0,28
Fate Sees_170	<0,5	n.a.
Fate Sees_171	<0,5	n.a.
Fate Sees_175	0,80	n.a.
Fate Sees_177	17,9	n.a.
Fate Sees_179	<0,5	n.a.
Fate Sees_180	<0,5	n.a.
Fate Sees_181	<0,5	n.a.
Fate Sees_182	<0,5	n.a.
Fate Sees_183	0,74	n.a.
Fate Sees_184	<0,5	n.a.
Fate Sees_189	0,6	n.a.
Fate Sees_190	<0,5	n.a.
Fate Sees_191	<0,5	n.a.
Fate Sees_193	<0,5	n.a.
Fate Sees_194	<0,5	0,12

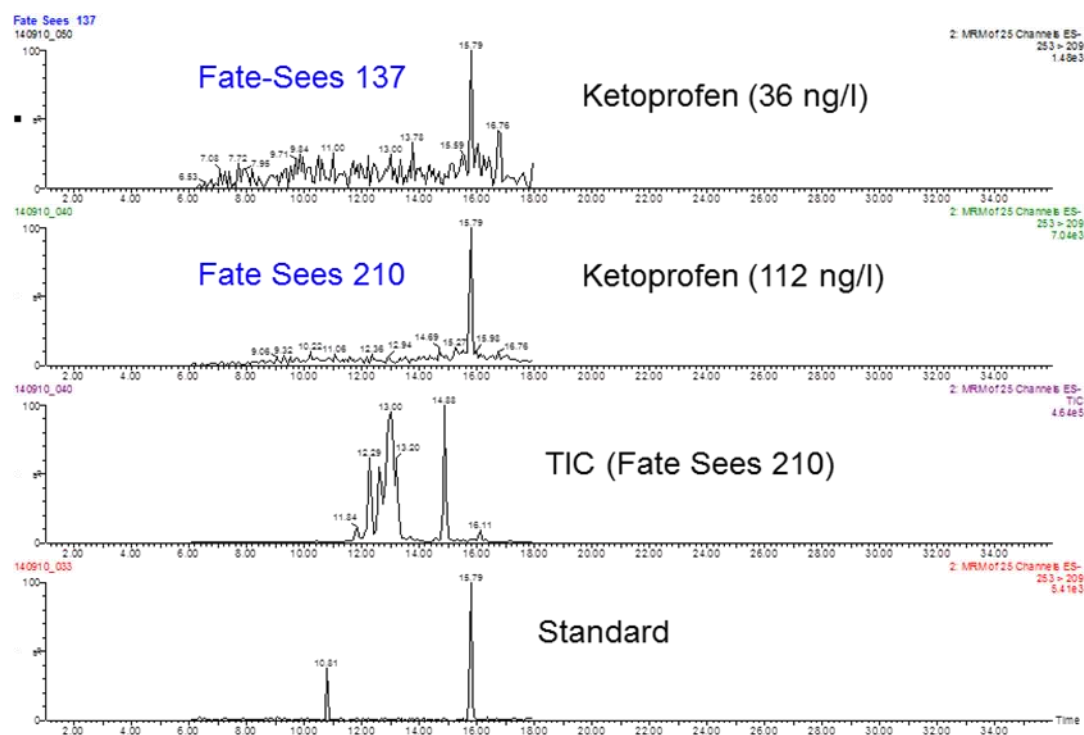
Fate Sees_195	<0,5	0,32
Fate Sees_196	<0,5	0,20
Fate Sees_198	<0,5	n.a.
Fate Sees_199	1,23	n.a.
Fate Sees_202	<0,5	n.a.
Fate Sees_203	<0,5	0,10
Fate Sees_204	0,53	0,10
Fate Sees_205	<0,5	0,11
Fate Sees_208	<0,5	0,26
Fate Sees_209	3,27	0,21
Fate Sees_210	<0,5	0,44
Fate Sees_211		
Fate Sees_214		
Fate Sees_226	12,21	<0,1
Fate Sees_227	<0,5	<0,1
Fate Sees_228	0,97	0,15
Fate Sees_229	<0,5	0,12
Fate Sees_233	<0,5	n.a.
Fate Sees_235	<0,5	n.a.
Fate Sees_236	<0,5	0,14
Fate Sees_239	0,94	n.a.
Fate Sees_242	<0,5	n.a.
Fate Sees_243	<0,5	n.a.
Fate Sees_245	<0,5	n.a.
Fate Sees_247	<0,5	n.a.
Fate Sees_248	0,59	0,16
Fate Sees_250	<0,5	n.a.
Fate Sees_252	<0,5	n.a.
Fate Sees_253	0,6	n.a.
Fate Sees_254	6,0	n.a.
Fate Sees_256	0,84	n.a.
Fate Sees_257	<0,5	n.a.
Fate Sees_258	<0,5	n.a.
Fate Sees_259	0,67	n.a.
Fate Sees_260		
Fate Sees_261		
Fate Sees_262		
Fate Sees_263		
Fate Sees_264		
Fate Sees_265		
Fate Sees_266		
Fate Sees_267		
Fate Sees_268		

Annex 10: Analytical results for inorganic elements in WWTP effluents [mg/l]. Lab: JRC-IES. Be and Cr were in all cases below the LOD (0.001 mg/l).

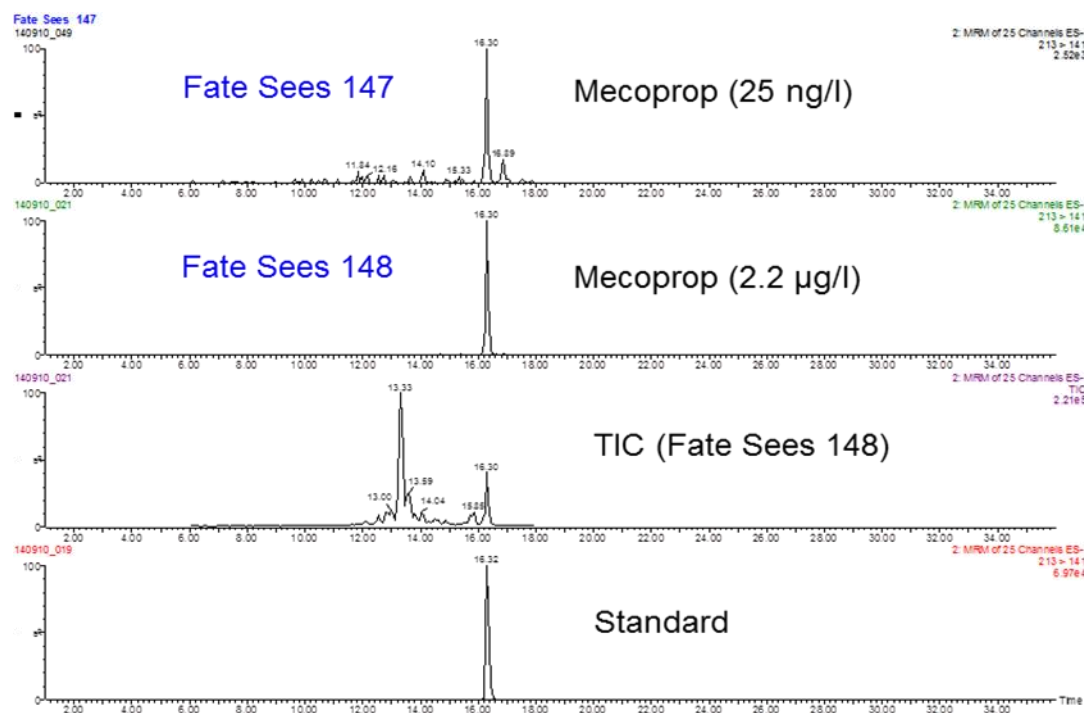
n.d. means below LOD.

Sample	Hg	Ag	Al	As	Ba	Cd	Co	Cu	Mg	Mn	Mo	Ni	Pb	Sb	Se	Tl	Zn
LOD [mg/l]	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
Fate Sees_136	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	89,2	0,10	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_137	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	69,2	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_138	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	8,2	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_139	n.d.	n.d.	n.d.	n.d.	n.d.	0,04	n.d.	n.d.	6,1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_140	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9,1	0,01	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,10
Fate Sees_141	n.d.	n.d.	n.d.	n.d.	n.d.	0,01	n.d.	n.d.	7,6	0,08	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_142	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9,1	0,04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,06
Fate Sees_143	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	23,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_144	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_145	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	16,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_146	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	28,1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_147	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	19,8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_148	n.d.	n.d.	n.d.	n.d.	n.d.	0,03	n.d.	n.d.	21,2	0,32	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_149	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	15,3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_150	n.d.	n.d.	0,11	n.d.	n.d.	n.d.	n.d.	n.d.	25,4	0,08	0,27	0,16	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_151	n.d.	n.d.	n.d.	n.d.	0,05	n.d.	n.d.	n.d.	55,4	0,35	0,50	0,05	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_152	n.d.	n.d.	0,58	n.d.	n.d.	n.d.	0,06	n.d.	10,2	0,13	0,09	0,42	n.d.	0,90	n.d.	n.d.	0,24
Fate Sees_153	n.d.	n.d.	0,13	n.d.	n.d.	n.d.	n.d.	0,03	15,1	0,07	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,15
Fate Sees_155	n.d.	n.d.	n.d.	n.d.	0,04	n.d.	n.d.	n.d.	36,5	0,49	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_156	n.d.	n.d.	0,13	n.d.	0,03	n.d.	n.d.	n.d.	0,1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Fate Sees_157	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	61,5	0,15	0,18	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_158	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	20,5	0,07	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,06
Fate Sees_159	n.d.	n.d.	0,20	n.d.	0,04	n.d.	n.d.	n.d.	10,7	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_160	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7,5	0,04	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	0,01
Fate Sees_161	n.d.	n.d.	0,30	n.d.	n.d.	n.d.	n.d.	n.d.	57,0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,009
Fate Sees_162	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	33,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,007
Fate Sees_163	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	67,4	0,04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Fate Sees_164	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	n.d.	30,3	0,09	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	19,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_166	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	8,3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_167	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	12,8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_168	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6,3	0,03	0,10	n.d.	n.d.	n.d.	n.d.	n.d.	0,09
Fate Sees_169	n.d.	n.d.	n.d.	n.d.	0,01	n.d.	n.d.	n.d.	19,5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,09
Fate Sees_170	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9,2	0,19	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_171	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	10,7	0,14	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_175	n.d.	n.d.	n.d.	n.d.	0,05	n.d.	n.d.	n.d.	83,0	0,07	0,01	n.d.	n.d.	n.d.	n.d.	n.d.	0,01
Fate Sees_177	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	53,1	0,01	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_179	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	11,2	0,18	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_180	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	11,2	0,34	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_181	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7,0	0,05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_182	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_183	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	144,0	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_184	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	n.d.	87,3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_189	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_190	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	n.d.	24,6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_191	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	14,0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,01
Fate Sees_193	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	14,8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_194	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	15,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_195	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	23,8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,06
Fate Sees_196	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7,0	0,02	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_198	n.d.	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	11,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_199	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	13,6	0,08	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,01

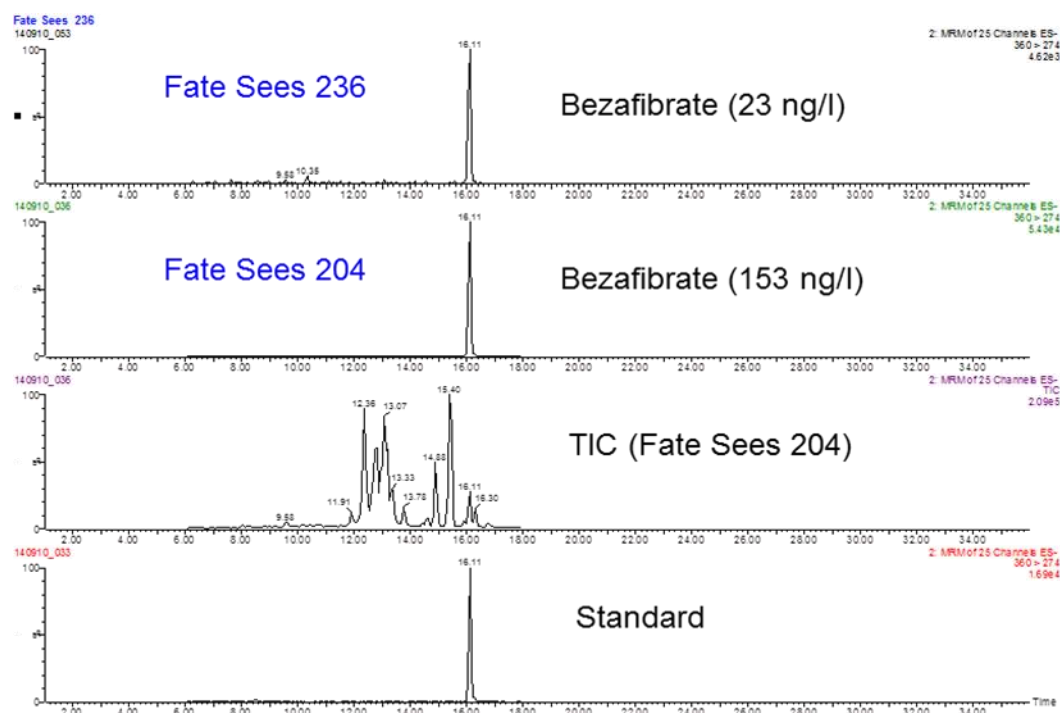
Fate Sees_202	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	17,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,06
Fate Sees_203	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	28,6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_204	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	19,8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_205	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03	12,5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_208	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	24,5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_209	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	29,0	0,02	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_210	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	15,0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,05
Fate Sees_211	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_214	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7,2	0,006	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_226	n.d.	n.d.	n.d.	n.d.	n.d.	0,04	n.d.	n.d.	16,0	0,05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_227	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	13,7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_228	n.d.	n.d.	n.d.	n.d.	n.d.	0,01	n.d.	n.d.	14,4	0,02	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,14
Fate Sees_229	n.d.	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	30,0	0,01	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,01
Fate Sees_233	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	10,8	0,13	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_235	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	14,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_236	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	12,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_239	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	12,1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,07
Fate Sees_242	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	8,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,5	0,13	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_245	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7,8	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,03
Fate Sees_247	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	13,6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_248	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,2	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,01
Fate Sees_250	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	10,3	0,05	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_252	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	18,4	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_253	n.d.	n.d.	0,12	n.d.	n.d.	n.d.	n.d.	n.d.	8,3	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,09
Fate Sees_254	n.d.	n.d.	0,05	n.d.	n.d.	n.d.	n.d.	n.d.	8,2	0,04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,04
Fate Sees_256	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	30,4	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_257	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6,3	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_258	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	14,9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,02
Fate Sees_259	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6,4	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0,01
Fate Sees_260	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,4	0,20	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_261	n.d.	n.d.	n.d.	n.d.	n.d.	0,009	n.d.	n.d.	4,9	0,08	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_262	n.d.	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	3,6	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_263	n.d.	n.d.	n.d.	n.d.	n.d.	0,01	n.d.	n.d.	6,5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_264	n.d.	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	7,9	0,11	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_265	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5,0	0,14	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_266	n.d.	n.d.	n.d.	n.d.	n.d.	0,02	n.d.	n.d.	10,6	0,03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_267	n.d.	n.d.	n.d.	n.d.	n.d.	0,008	n.d.	n.d.	6,8	0,005	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.
Fate Sees_268	n.d.	n.d.	n.d.	n.d.	n.d.	0,006	n.d.	n.d.	10,3	0,16	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.a.



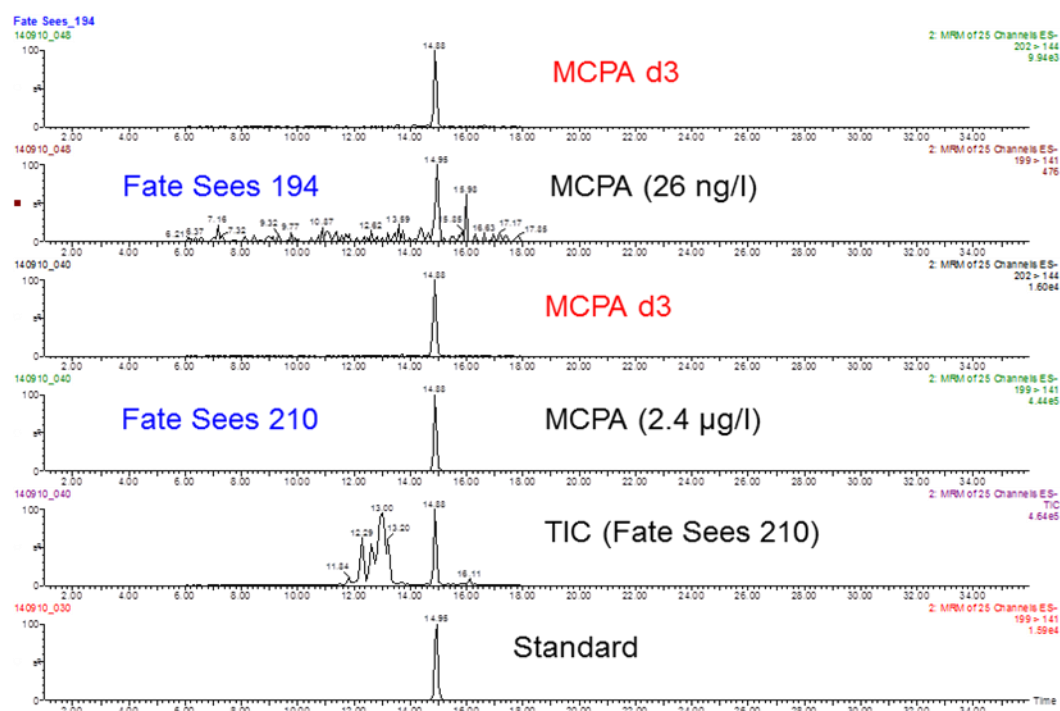
Annex Fig. 3: LC-MS² chromatograms of Ketoprofen in Fate Sees samples 137 (Cyprus), and 210 (Spain).



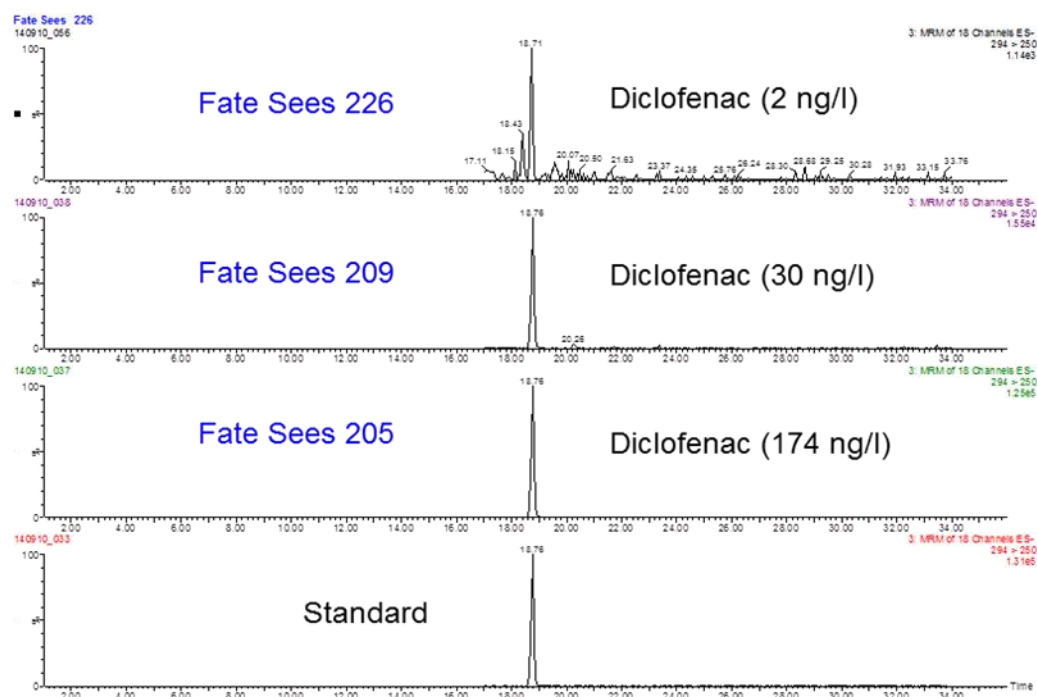
Annex Fig. 4: LC-MS² chromatograms of Mecoprop in Fate Sees samples 147 (CZ), and 148 (CZ).



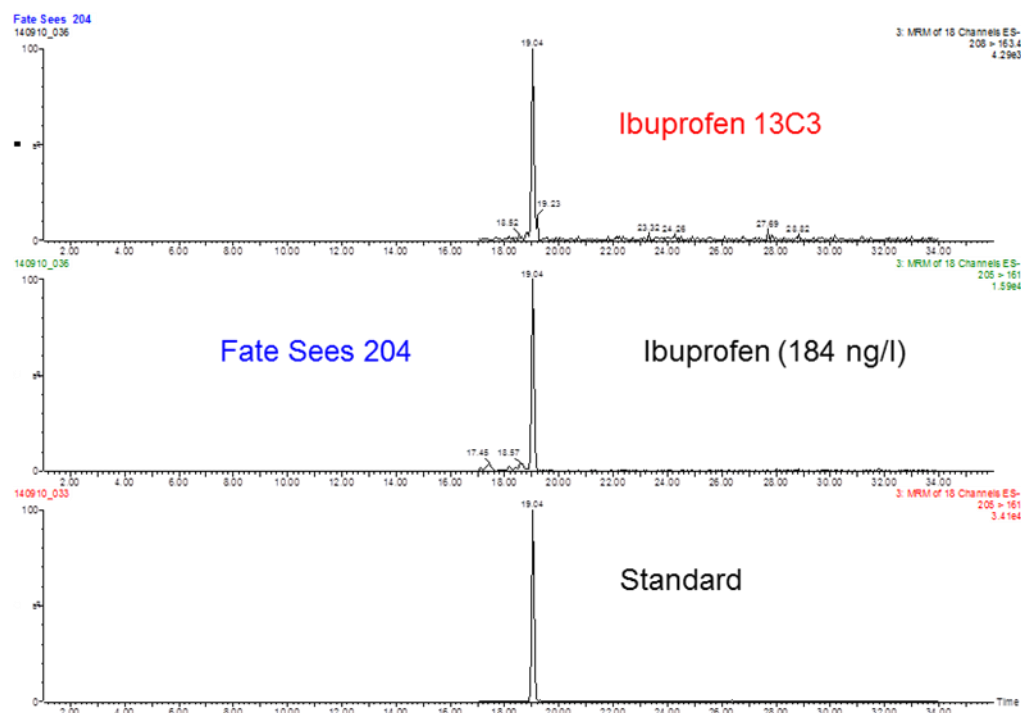
Annex Fig. 5: LC-MS² chromatograms of Bezaifibrate in Fate Sees samples 236 (NL), and 204 (Austria).



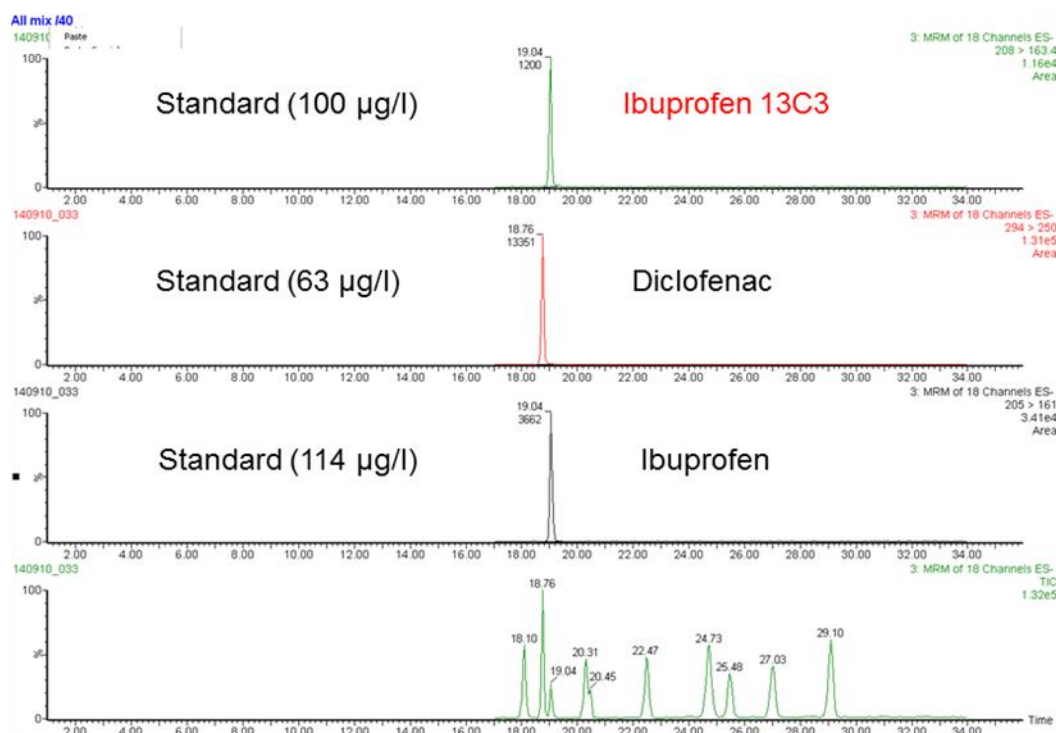
Annex Fig. 6: LC-MS² chromatograms of MCPA in Fate Sees samples 194 (CH), and 210 (Spain).



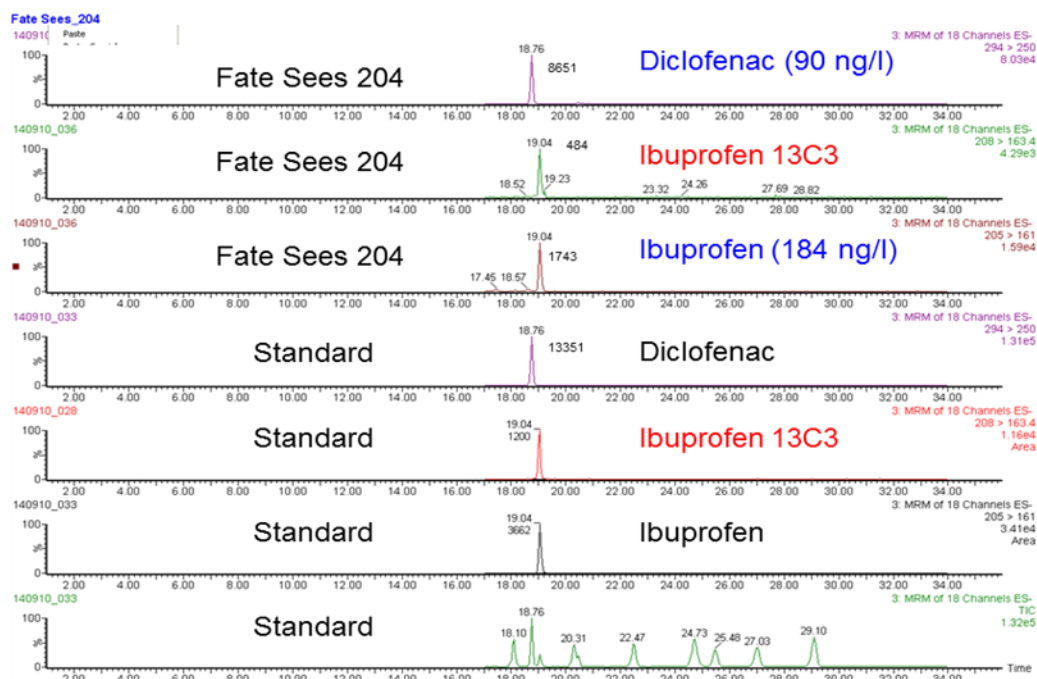
Annex Fig. 7: LC-MS² chromatograms of Diclofenac in Fate Sees samples 226 (Italy), 209 (Spain), and 205 (Austria).



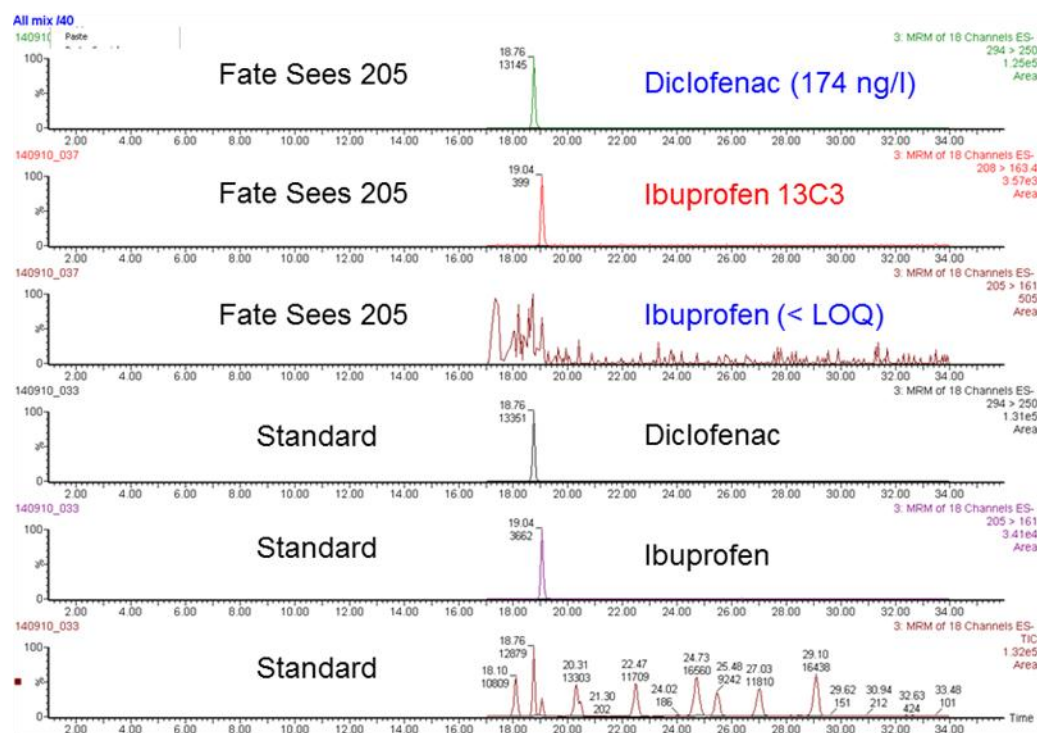
Annex Fig. 8: LC-MS² chromatograms of Ibuprofen in Fate-Sees sample 204 (Austria).



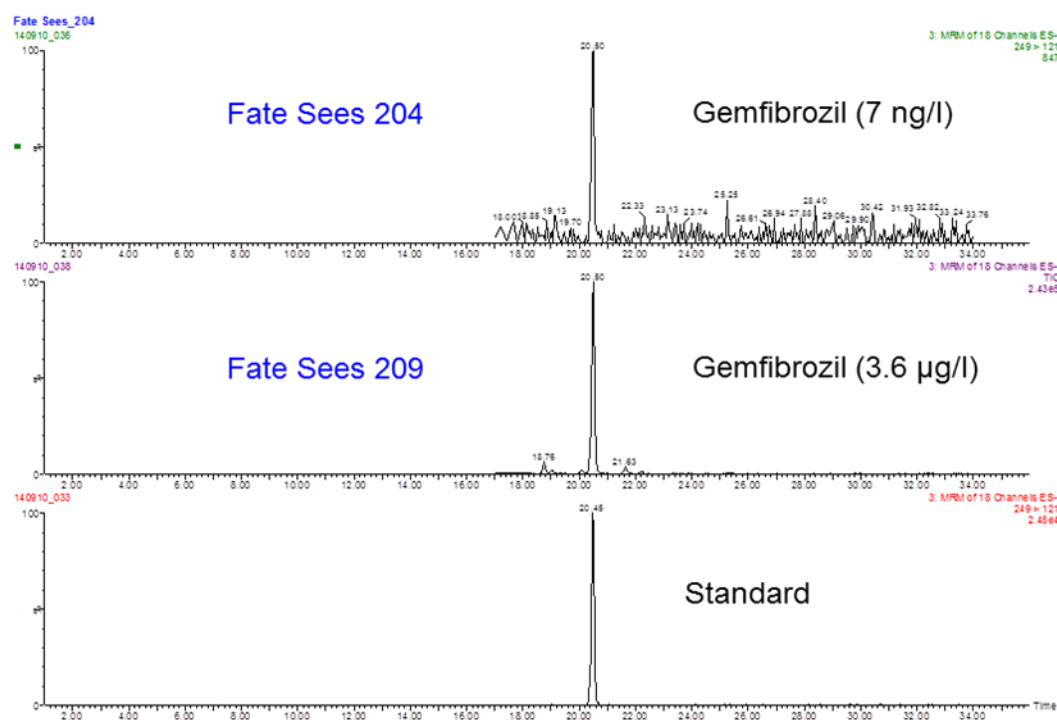
Annex Fig. 9: Analytical standards of Diclofenac and Ibuprofen.



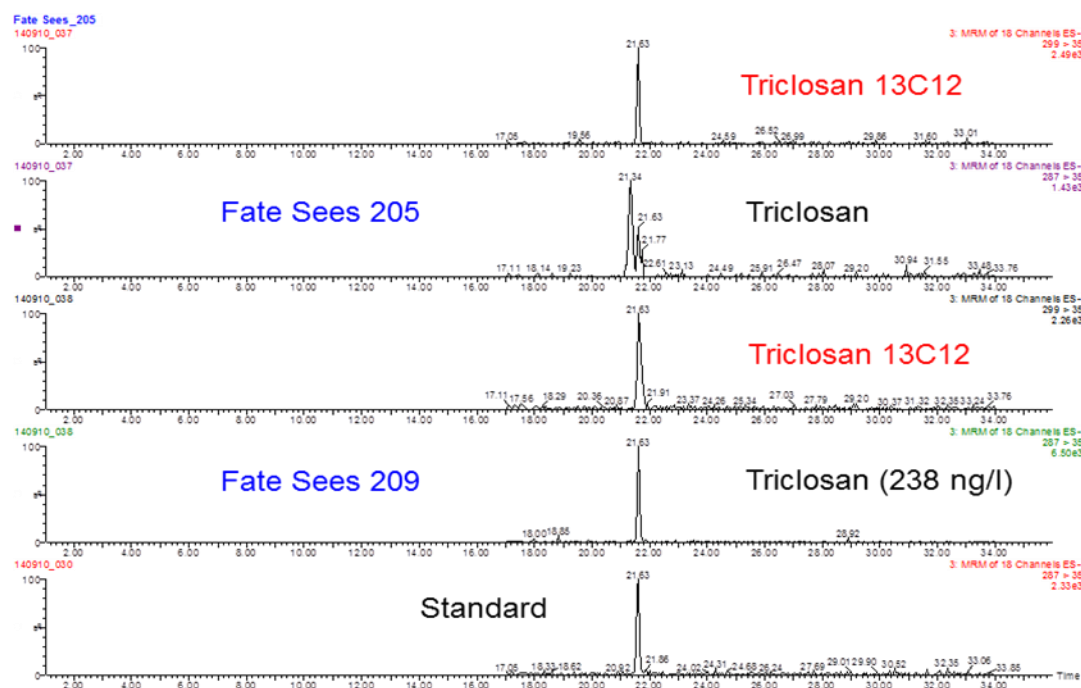
Annex Fig. 10: Fate Sees sample 204 (AWV Wiener Neustadt-Sud). Diclofenac and Ibuprofen together with standards.



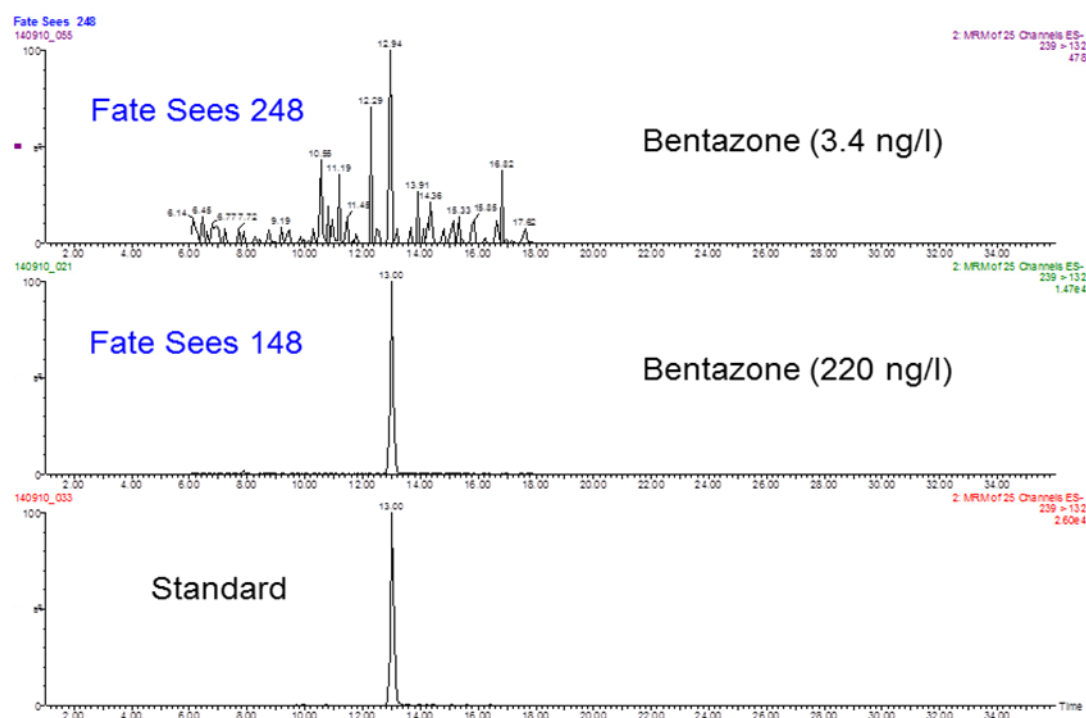
Annex Fig. 11: Fate Sees sample 205 (Wasserverband Ossiacher See). Diclofenac and Ibuprofen together with standards.



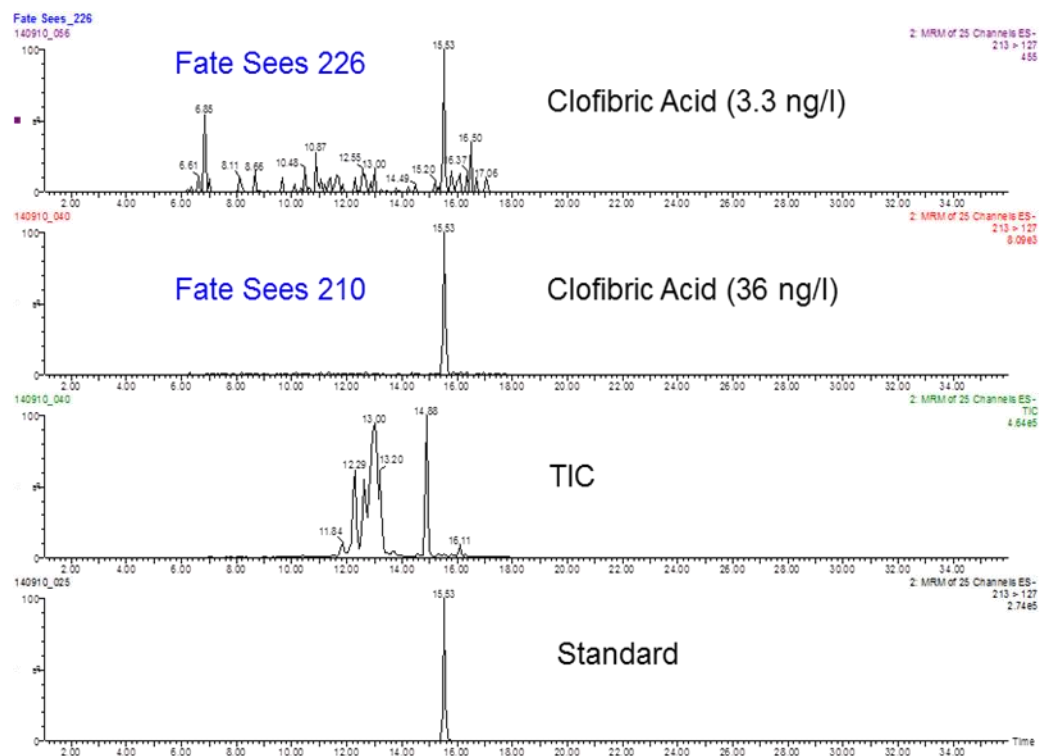
Annex Fig. 12: LC-MS² chromatograms of Gemfibrozil in Fate-Sees samples 204 (Austria), and 209 (Spain).



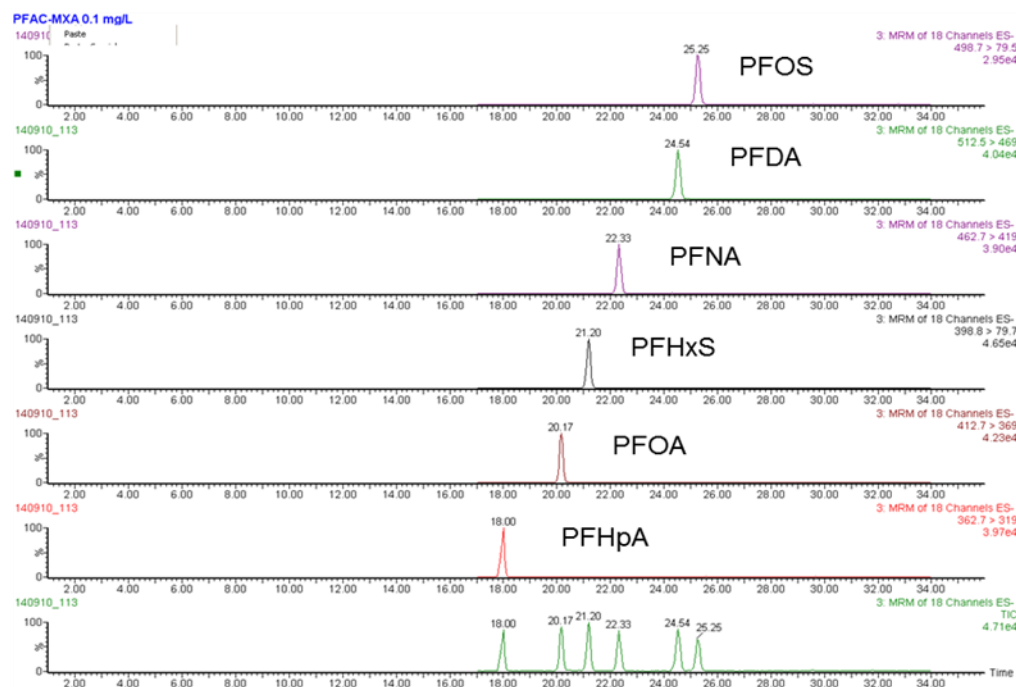
Annex Fig. 13: LC-MS² chromatograms of Triclosan in Fate-Sees samples 205 (Austria), and 209 (Spain).



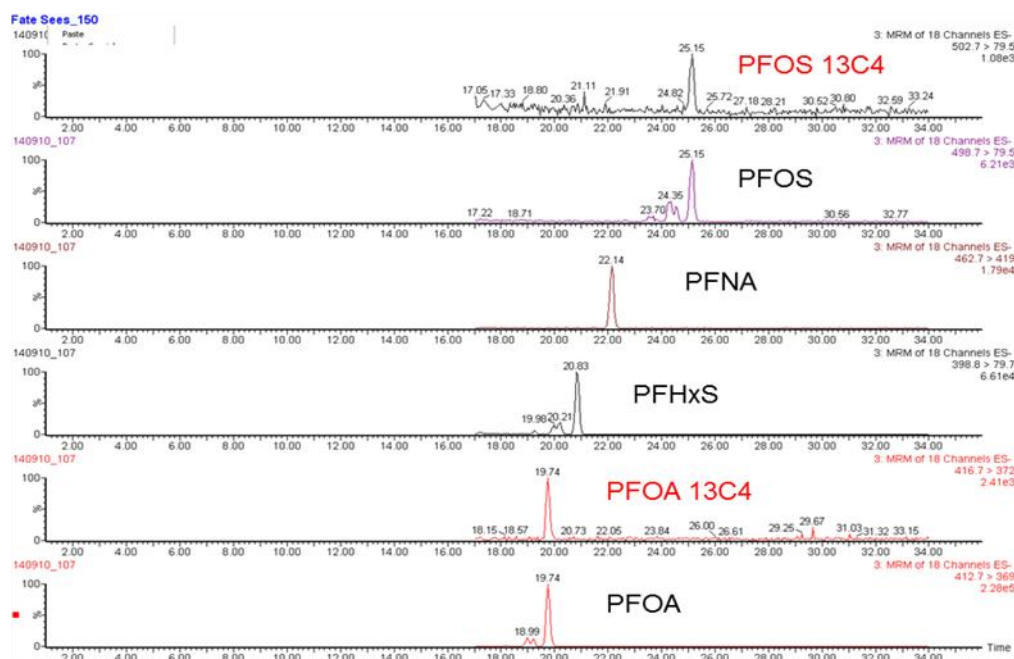
Annex Fig. 14: LC-MS² chromatograms of Bentazone in Fate-Sees samples 248 (NL), and 148 (CZ).



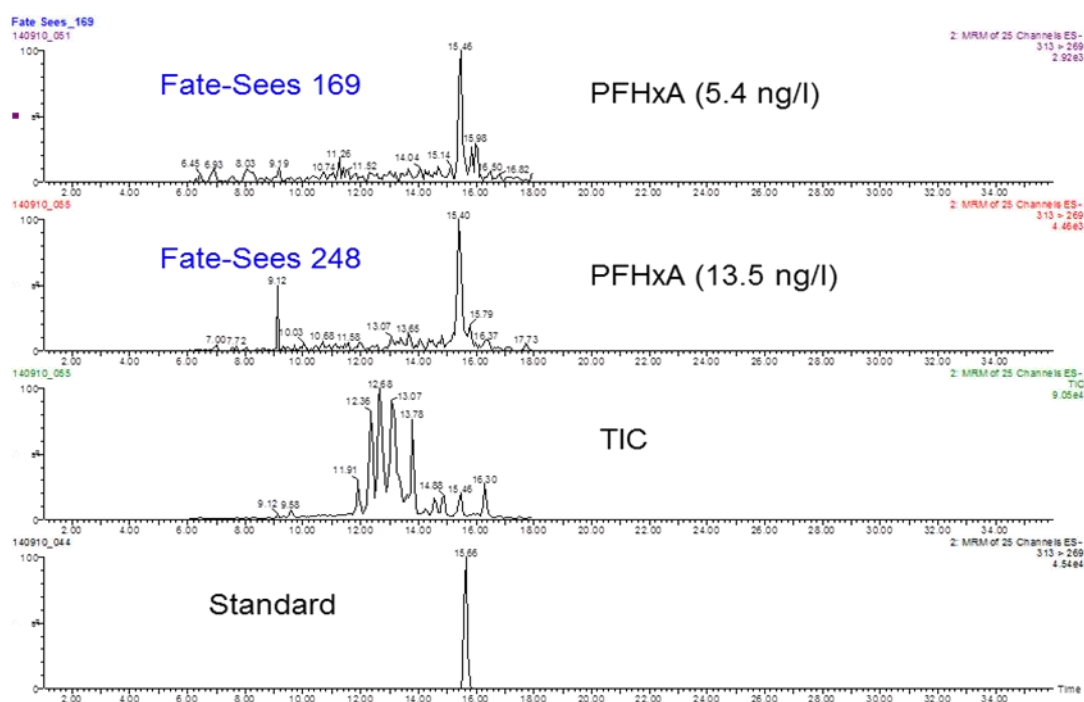
Annex Fig. 15: LC-MS² chromatograms of Clofibric acid in Fate-Sees samples 226 (Italy), and 210 (Spain).



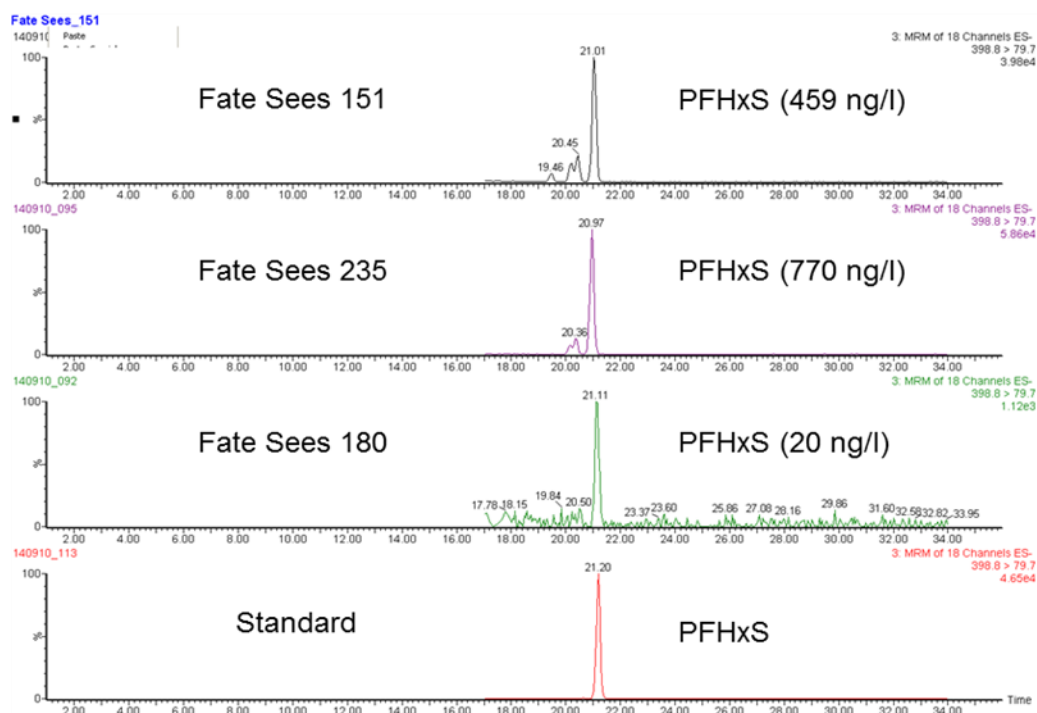
Annex Fig. 16: LC-MS² chromatograms of PFAS in Standard.



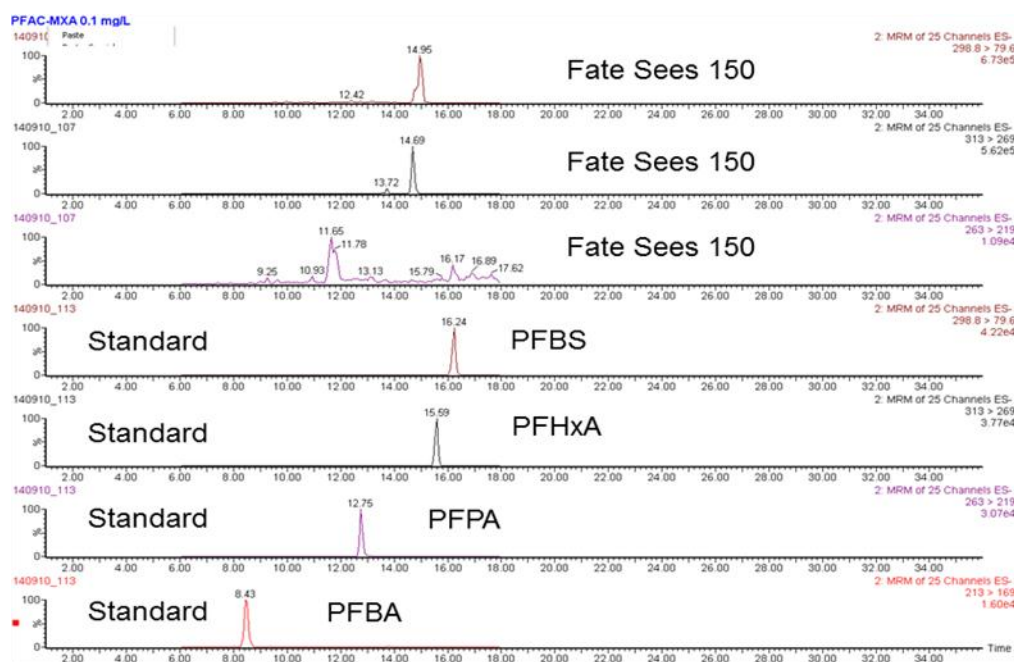
Annex Fig. 17: LC-MS² chromatograms of PFASs in Fate-Sees samples 150 (Belgium).



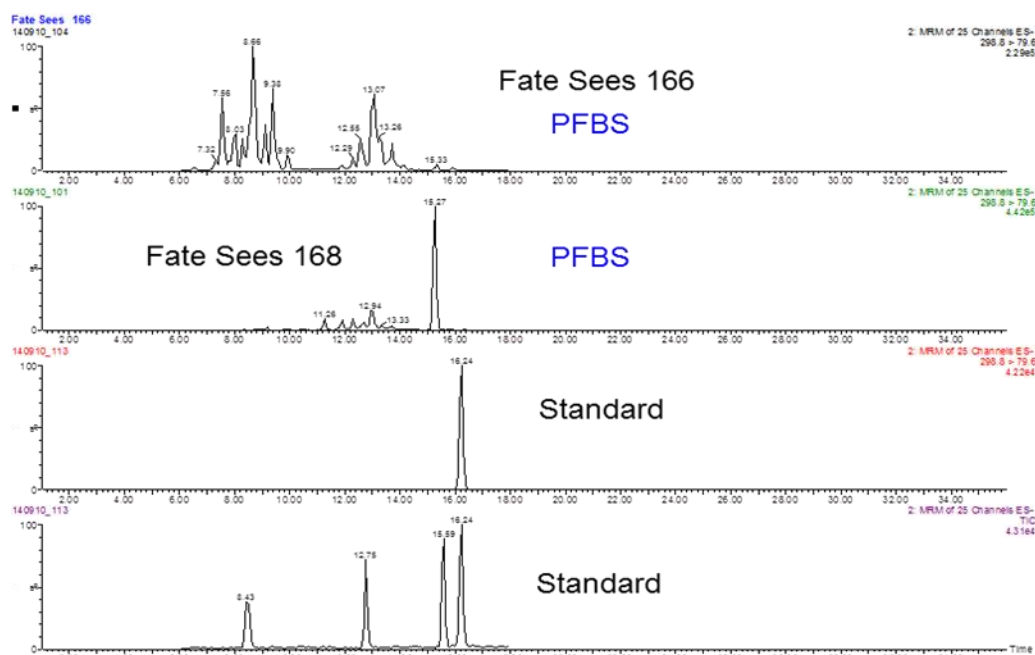
Annex Fig. 18: LC-MS² chromatograms of PFHxA in Fate-Sees samples 169 (Slovenia), and 248 (NL).



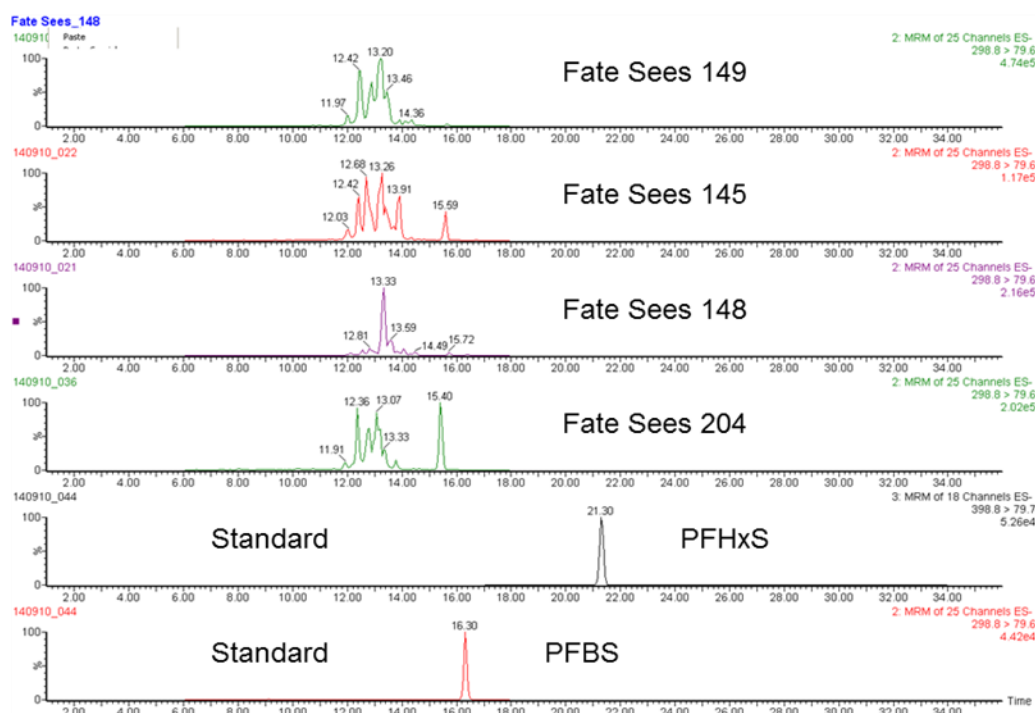
Annex Fig. 19: LC-MS² chromatograms of PFHxS in Fate-Sees samples 151 (Belgium), 235 (NL), and 180 (Finland).



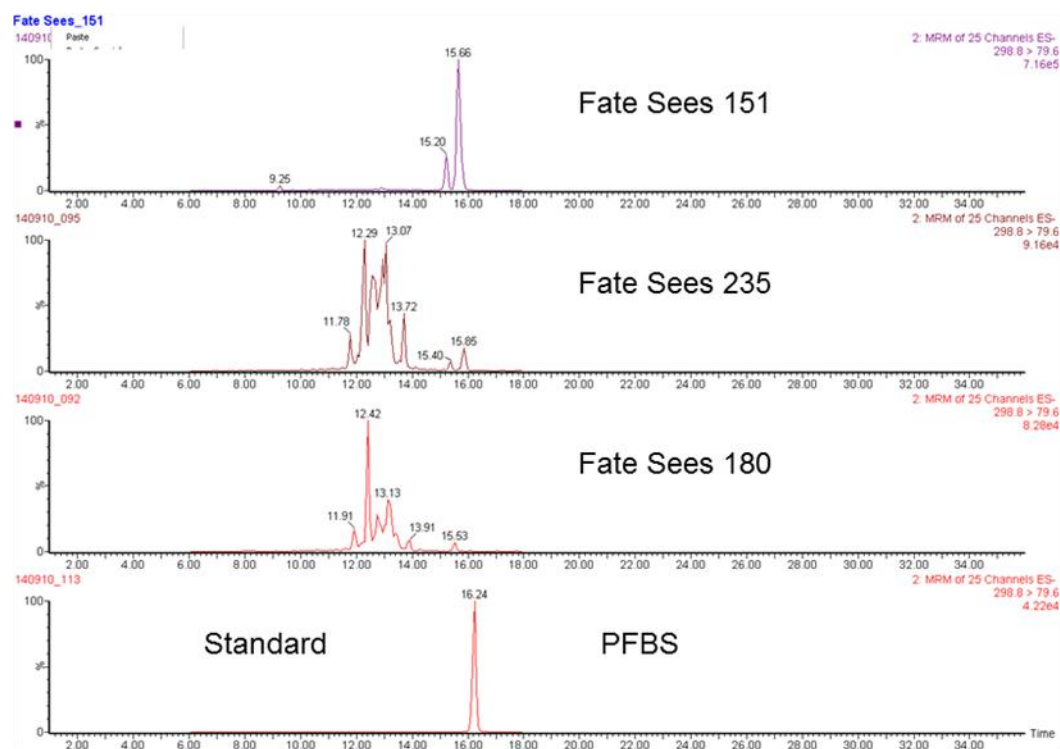
Annex Fig. 20 : LC-MS² chromatograms of small-chain PFASs in a standard solution and Fate Sees sample 150 (Belgium). No retention time match.



Annex Fig. 21: LC-MS² chromatograms of PFBS in Fate Sees samples 166 (Belgium) and 168 (Belgium). No retention time match with standard.



Annex Fig. 22: LC-MS² chromatograms of different Fate Sees samples for PFBS and PFHxS. No retention time match with standard.



Annex Fig. 23: LC-MS² chromatograms of different Fate Sees samples for PFBS. No retention time match with standard.

Title: EU Wide Monitoring Survey on Waste Water Treatment Plant Effluents

Authors: Robert Loos, Raquel Carvalho, Diana C. António, Ludek Blaha, Barbora Jarosova, Stefan Voorspoels, David Schwesig, Peter Haglund, Jerker Fick, Oliver Gans, Sara Comero, Michela Ghiani, Teresa Lettieri, Giovanni Locoro, Bruno Paracchini, Simona Tavazzi, Bernd M. Gawlik

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Abstract

In the year 2010, effluents from 90 European waste water treatment plants (WWTPs) were collected and analysed in total for 160 organic chemicals and 20 inorganic trace elements. The analyses were complemented by applying also effect-based monitoring approaches aiming at estrogenicity and dioxin-like toxicity analysed by in vitro reporter gene bioassays, and yeast and diatom culture acute toxicity optical bioassays. The analytical work was performed in six European expert laboratories.

Analyses of organic substances were performed by solid-phase extraction (SPE) or liquid-liquid extraction (LLE) followed by high performance liquid chromatography tandem mass spectrometry (HPLC-MS-MS) or gas chromatography high-resolution mass spectrometry (GC-HRMS). Inorganic compounds were analysed by inductively coupled plasma – mass spectrometry (ICP-MS) or ICP atomic emission spectrometry (ICP-AES).

This European-wide monitoring study on the occurrence of micropollutants in WWTP effluents represents the largest EU wide monitoring survey on WWTP effluents ever performed. It produced a comprehensive data set on many so far only locally investigated “emerging” compound classes including pharmaceuticals and personal care products (PPCPs), veterinary (antibiotic) drugs, perfluoroalkyl substances (PFASs), organophosphate ester flame retardants, pesticides (and some metabolites) or industrial chemicals such as benzotriazoles (corrosion inhibitors), polycyclic musk fragrances, x-ray contrast agents, Gadolinium compounds, and siloxanes.

The obtained results show the presence of 131 target organic compounds in European wastewater effluents, in concentrations ranging from low nanograms to milligrams per liter. These results obtained from 90 different European WWTPs allow the calculation of a European median level for the chemicals investigated. The most relevant compounds identified in the effluent water samples in terms of frequency of detection, maximum, average and medium concentration levels were Sucralose, Acesulfame K (artificial sweeteners), PFOA, PFHxA, PFHpA, PFOS (perfluoroalkyl substances), DEET (insect repellent), Benzotriazoles (corrosion inhibitors), the pharmaceuticals Bisoprolol, Carbamazepine, Ciprofloxacin, Citaprolam, Clindamycin, Codeine, Diltiazem, Diphenhydramin, Eprosartan, Fexofenadine, Flecainide, Gemfibrozil, Fluconazole, Haloperidol, Ibuprofen, Ketoprofen, Oxazepam, Risperidone, Sulfamethoxazole, Telmisartan, Tramadol, Trimethoprim, Venlafaxin, the organophosphate ester flame retardants Tri-iso-butylphosphate (TIBP), Tributylphosphate (TBP), Tris(2-chloroethyl)phosphate (TCEP), Tris(2-chloroisopropyl)phosphate (TCPP), Tris(2-butoxyethyl)phosphate (TDCP), Tris(2-butoxyethyl)phosphate (TBEP), Triphenyl-phosphate (TPP), 2-Ethylhexyldiphenyl-phosphate (EHDPP), the x-ray contrast media Amidotrizoic acid, Iohexol, Iopromid, Iomeprol, Iopamidol, the pesticides Terbutylazine, Terbutylazine-desethyl (metabolite), MCPA, Mecoprop, Diuron, Triclosan (antibacterial), and Gadolinium (from magnetic resonance imaging contrast media used in hospitals).

The toxicity tests applied showed some toxicity. From the total number of 75 WWTP effluents analyzed for estrogenicity, 27 sample extracts showed estrogenic activity higher than the detection limit > 0.5 ng/l estrogen equivalents (EEQ). Twenty five effluent sample extracts were screened for dioxin-like activity, and twenty one out of these 25 tested sample extracts exceeded the detection limit (0.1 ng/l TEQ_{bio-test}) but the maximal detected dioxin-like activity was only 0.4 ng/l TEQ_{bio-test}. Finally, three out of 13 effluent samples tested on acute toxicity revealed themselves harmful for the growth of yeast and diatom organisms.

Finally, the results were compared with other published local, national, or smaller international studies on WWTPs. This comparison showed that lower median effluent concentrations were found in our large-scale European-wide study ($n=90$) compared to local studies of single WWTPs. It is difficult to compare results of median concentrations of one WWTP (or a small number) with the median of this study ($n=90$) because many different plants around Europe were included. In contrast, maximum and average concentrations are in relatively good agreement to other studies.

What is clear is that the elimination of most of the anthropogenic substances in conventional WWTPs with secondary biological treatment is incomplete and improvements of wastewater treatment and subsequent treatments of the produced sludge are required to prevent the introduction of these micropollutants in the environment. It must be considered that today's conventional waste water treatment technology (mechanical and biological steps) is from the 1970s; it was designed to remove nitrogen and phosphorus, and some non-polar chemicals which are removed with the sewage sludge.

It is being discussed in Europe to upgrade WWTPs with additional tertiary treatment steps such as ozonation and/or powdered activated carbon adsorption to remove micropollutants from WWTP effluents. In the Swiss “Micropoll Strategy” project complementary treatment steps have been evaluated and it has been shown that water quality can be significantly improved using processes such as powdered activated carbon adsorption or ozonation.

Under the view of escalating population growth, and increased water stress in many regions of the world, reuse of treated water and waste water recycling are becoming more important options for water supply. The increasing worldwide contamination of freshwater systems with thousands of industrial and natural chemical compounds is one of the key environmental problems facing humanity. Although most of these compounds are present at low concentrations, many of them raise considerable toxicological concerns, particularly when present as components of complex mixtures (Schwarzenbach et al., 2006).

As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

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